

U. S. Department of Transportation Research and Special Programs Administration

Property Specific Sampling and Analysis Plan,
Air and Dust Sampling
for
Stimson Lumber Company
Libby Asbestos Project
Libby, Montana

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Acronyms

AHERA Asbestos Hazard Emergency Response Act
ASTM American Society for Testing Materials

BZ breathing zone

CFR Code of Federal Regulations

COC chain-of-custody EL excursion limit

EPA U. S. Environmental Protection Agency

IAW in accordance with

ISO International Organization for Standardization

L/min liters per minute MCE mixed cellulose ester

NIOSH National Institute for Occupational Safety and Health

OSHA Occupational Safety and Health Administration

PCM phase contrast microscopy

QA quality assurance QC quality control

RPM remedial project manager SAP sampling and analysis plan

SL Stimson Lumber

SOP standard operating procedure
TEM transmission electron microscopy

TWA-PEL time weighted average-permissible exposure limit

cm² square centimeter

μm micrometer

Property Specific Sampling and Analysis Plan, Air and Dust Sampling, Stimson Lumber Company Libby, Montana Libby Asbestos Project

November 2001 (revised August 2002)

1.0 Objective

The Stimson Lumber Company property is located in the eastern section of Libby, Montana, on Highway 2 South. Historical Information regarding the property suggests that numerous vermiculite products and wastes were used at, or transported to, the property at various times and at various locations. Much of this material is still present. Additionally, a vermiculite processing plant was located on the property until approximately 1950. It is believed that these products contain varying levels of the Libby Amphibole asbestiform. The U.S. Environmental Protection Agency (EPA) has already determined that materials originating from the Libby vermiculite mine can act as sources of Libby Amphibole asbestiform and should be removed (EPA, 2002). Therefore current investigations at "typical" residential and commercial properties in Libby focus on finding these sources. However, because of the complexity of the Stimson facility relative to most properties in Libby, less is known about potential exposures, if any, that may result from disturbances of these source materials. Therefore, a more comprehensive investigation will be required at this facility than at other less complex commercial properties in Libby. To investigate the potential impacts, EPA is pursuing a two part approach. First, air and dust samples will be collected as described in this Sampling and Analysis Plan (SAP) in areas where vermiculite is known or suspected to be present. This will provide potential exposure information in these areas. Second, a separate study will be conducted later this summer to screen all areas of the facility for potential Libby Amphibole asbestiform sources, similar to screenings of all properties in Libby being conducted this year. The primary objective of that work is to document and delineate potential sources of Libby Amphibole asbestiform in a comprehensive, systematic manner. A separate planning document will be prepared for that effort, based on the Contaminant Screening Study SAP (CDM 2002) recently developed. The two parts of the study will ultimately be combined, summarized in a specific report, and used to (1) determine the need for any immediate actions, (2) determine the need for cleanup, (3) prioritize any future cleanup activities across the facility, and (4) determine the extent of contamination and delineate areas shown to be clean.

2.0 Air Sampling

2.1 Personal Air Sampling

2.1.1 Selecting Sample Locations

Personal breathing zone (BZ) samples will be collected on a total of nine Stimson Lumber Company employees using a task-based, i.e., job function, approach. Personal BZ samples will be collected in accordance with (IAW) EPA Standard Operating Procedure (SOP) 2015 Asbestos Sampling (Appendix A). Volume requirements will be IAW the Occupational Safety and Health Administration (OSHA) Construction Standard for Asbestos, 29 Code of Federal Regulations (CFR) 1926.1101. Full work shift, time weighted average-permissible exposure limit (TWA-PEL) samples will be collected on the following employees:

- Five workers in the Plywood Plant (one for each separate job function, i.e., dryer tender, dryer feeder, dryer offbearer, plugger, and green chain)
- Two workers in the Central Maintenance Building
- One worker in the Fingerjoint Building
- One worker in the Log Yard

Representative excursion limit (EL) sampling will also be conducted to document potential worker exposure to airborne concentrations of asbestos as averaged over a sampling period of 30 minutes. These 30-minute EL measurements will be associated with those lumber company operations, job functions and work practices that are most likely to produce exposures above the excursion limit for employees in each work area.

These nine employees will be sampled for three consecutive days. Sampling cassettes will be visually inspected approximately every 2 hours for filter darkening. If loose dust is seen accumulating on the filter, indicating potential filter membrane overloading, the sampling cassettes shall be immediately changed out. The total representative number of personal BZ samples collected for both PEL and EL evaluations will be determined during a preliminary site visit and be based on company operations, work practices, and job functions. The estimated total number of PEL and EL samples will also be agreed upon by the EPA remedial project manager (RPM) and the Volpe Center representative.

2.1.2 Collection and Analysis of Personal Air Samples

Personal BZ air samples will be collected by drawing air through a mixed cellulose ester (MCE) filter (0.8 micrometer [μ m] pore size) at a specified flow rate during the work shift. The details of the collection method are provided in EPA SOP 2015 (Appendix A). Personal air samples will be collected at a flow rate between 0.5 to 2.5 liters per minute (L/min) for the 25-millimeter cassette. It is presently anticipated that sampling cassettes may have to be changed out every 4 hours. Several employees in the Stimson Lumber Company work shifts longer than 8 hours. For example, employees

working on the "dryer" process often work 12-hour shifts, and "green chain" workers may work 10-hour shifts. For these employees, the measured exposure level for the entire work shift will be adjusted to the 8-hour TWA-PEL for these unusual work schedules. The measured concentration without respect to sample duration (e.g., "raw" data) will also be reported.

All personal air samples will be analyzed IAW International Standard - Determination of Asbestos Fibers (International Organization for Standardization [ISO] 10312), National Institute for Occupational Safety and Health (NIOSH) Method 7400 - Asbestos by Phase Contrast Microscopy (PCM), and EPA Asbestos Hazard Emergency Response Act (AHERA) Transmission Electron Microscopy (TEM) analytical methods.

2.1.3 Quality Control Personal Air Samples

Field personnel will prepare two types of quality control (QC) samples, field blanks and lot blanks.

Field Blanks

Each set of samples taken will include 10 percent field blanks or a minimum of two field blanks. These blanks will come from the same lot as the filters used for the sample collection. The field blank results shall be averaged and subtracted from the analytical results before reporting. Any samples represented by a field blank having a fiber count in excess of the detection limit of the method being used shall be rejected. Field Blanks are collected by removing the cap from the sample cassette for not more than 30 seconds and replacing it at the time of sampling before sampling is initiated.

Lot Blanks

As a means of ensuring quality control in the sampling filter cassettes, field personnel will prepare a lot blank for personal air samples by labeling an unused filter cassette from the lot of cassettes used for the sampling and submit the cassette for analysis. Lot blanks will be examined to determine the background asbestos structure concentration.

2.2 Ambient Air

2.2.1 Selecting Sample Locations

Ambient air samples will be collected from three of the buildings at the Stimson Lumber Company (the Plywood Plant, Central Maintenance Facility, and the Finger Jointer Building) and two outdoor areas (the employee parking lot near the former popping plant and the log yard). Approximately four ambient air samples will be collected from each of the buildings and the outdoor areas during normal daily operations and while equipment is running. In addition, one field replicate sample will be collected at each site. Therefore, an estimated total of 20 ambient air samples will be collected (excluding blanks). The specific locations of the ambient air sampling stations will be determined during the preliminary onsite visit. The EPA RPM, and the Volpe representative will concur with the locations of the ambient air samples to be collected in each building/area prior to sampling.

2.2.2 Collection and Analysis of Ambient Air Samples

Ambient air samples will be collected by drawing air through an MCE filter (0.8 μ m pore size). The details of the method are provided in EPA SOP 2015 Asbestos Sampling (Appendix A). Ambient air samples will be collected at a flow rate of 10 L/min over a 7- to 8-hour time period.

All ambient air samples will be analyzed IAW International Standard - Determination of Asbestos Fibers (ISO 10312) NIOSH Method 7400, Asbestos by PCM, and EPA Asbestos Hazard Emergency Response Act (AHERA) Transmission Electron Microscopy (TEM) analytical methods.

2.2.3 Quality Control Ambient Air Samples

Field personnel will prepare and collect three types of QC samples, field blanks, lot blanks, and replicate samples.

Field Blanks

Each set of samples taken will include 10 percent field blanks or a minimum of two field blanks. These blanks will come from the same lot as the filters used for the sample collection. The field blank results shall be averaged and subtracted from the analytical results before reporting. Any samples represented by a field blank having a fiber count in excess of the detection limit of the method being used shall be rejected. Field blanks are collected by removing the cap from the sample cassette for not more than 30 seconds and replacing it at the time of sampling before sampling is initiated.

Lot Blanks

As a means of ensuring quality control in the sampling filter cassettes, field personnel will prepare a lot blank for ambient air samples by labeling an unused filter cassette from the lot of cassettes used for the sampling and submit the cassette for analysis. Lot blanks will be examined to determine the background asbestos structure concentration.

Replicate Samples

As a means of assessing sample variability during ambient air sampling, field personnel will collect field replicate samples (side-by-side samples). Field replicate samples will be collected at a rate of 1 per 10 samples (10 percent), or a minimum of one from each specific sampling location (a building, an outside area, etc.).

3.0 Microvacuum Dust Sampling

3.1 Selecting Sample Locations

Microvacuum dust samples will be collected within all of the buildings at the Stimson Lumber facility. One dust sample will be collected in each building that does not contain vermiculite. High traffic areas will be sampled to determine if outside sources of contamination may be present. These samples will be composite samples consisting of 3 subsamples per composite. Up to five dust samples will be collected from the

Plywood Plant and Central Maintenance building known to contain vermiculite insulation. Samples will be collected from horizontal surfaces (e.g., surfaces most likely to accumulate dust) within each building to adequately characterize any potential contamination. These samples will be composite samples consisting of 3 subsamples per composite.

The total microvacuum dust samples collected during this assessment will depend on the number of buildings identified potentially containing vermiculite. The specifics of the dust sampling locations will be determined during the preliminary site visit. The EPA RPM and Volpe representative working with the sampling team will determine the locations and number of dust samples to be collected in each building.

3.2 Collection and Analysis of Dust Samples

Microvacuum dust samples will be collected by drawing air through an MCE filter (0.45 µm pore size) at a flow rate of 2.0 L/min for a minimum sampling time of 2 minutes or until all visible dust or particulate matter has been removed from the sampling area, whichever comes first. The details of the method are provided in American Society for Testing Materials (ASTM) Standard D-5755-95, Standard Test Method for Microvacuum Sampling and Indirect Analysis Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations (Appendix B).

The following modification is noted:

Section 8.7, Sampling Area – The ASTM method indicates that a 100 square centimeter (cm²) sampling area be vacuumed per cassette. In order to obtain a more representative dust sample from several areas within each building, three separate 100-cm² sampling areas per sampling cassette will be vacuumed. Therefore, each cassette will represent the dust from a 300-cm² area versus a 100-cm² area.

All microvacuum dust samples will be analyzed IAW ISO 10312.

3.3 Quality Control Microvacuum Dust Samples

Field personnel will prepare two types of QC samples, field blanks and lot blanks.

Field Blanks

As a means of ensuring QC in the sampling filter cassettes, field personnel will prepare blank samples for dust samples by labeling unused filter cassettes from the same lot(s) and submitting them for analysis. Field blanks are collected by removing the cap from the sample cassette for not more than 30 seconds and replacing it at the time of sampling before sampling is initiated.

Lot Blanks

As a means of ensuring quality control in the sampling filter cassettes, field personnel will prepare a lot blank for microvacuum dust samples by labeling an unused filter

cassette from the lot of cassettes used for the sampling and submit the cassette for analysis. Lot blanks will be examined to determine the background asbestos structure concentration.

4.0 Sample Identification

Each personal air sample, ambient air sample and microvacuum dust sample will be identified with a unique coding system. For QC purposes, this coding system (Index ID) is designed to prevent accidental duplication of sample identification numbers and ensures that all samples have a unique identification number assigned to them. These codes start at SL-00001, which corresponds to the Stimson Lumber (SL) air/dust sampling team. The last five numbers are sequential so thousands of unique codes are available, as necessary. To ensure that the laboratory is "blind" or unbiased, and does not receive certain specific information about a sample, only the index identification code will be used to label sample cassettes.

This coding system may be modified to suit field conditions and any modifications will be clearly described in the applicable field logbook.

5.0 Sample Documentation

Sampling activities during this assessment will be documented in a field logbook and on field data sheets (Appendix C) to be maintained by the field team IAW CDM Federal Programs Corporation (CDM) SOP 4-1 Field Logbook Content and Control (Appendix D). The field team leader will be responsible for maintenance and document control of the field logbook.

6.0 Sample Custody, Packaging, and Shipping

This section details the sample custody and the classifying, identifying, labeling, packaging, and transportation of personal air samples collected during this investigation. Sample classification is necessary to ensure the protection of personnel involved in the shipment of samples, and to maintain the integrity of each sample. Personal air samples, ambient air samples, and microvacuum samples collected during this assessment will be packaged and shipped IAW CDM's SOP 2-1 Packaging and Shipping of Environmental Samples Revision 1, dated June 20, 2001 (Appendix E).

To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, chain-of-custody (COC) records will be used. The COC record will be maintained as physical evidence of sample custody and control and provides the means to identify, track, and monitor each individual sample from the point of collection through final data reporting. COC procedures will follow the requirements set forth in CDM SOP 1-2 Sample Custody, with modifications (Appendix F).

The following modifications to SOP 1-2 have been reviewed and approved:

Section 5.2, Sample Labels and Tags – A label will be affixed to each air sampling cassette prior to being shipped to the appropriate laboratory. This number will correspond to the number assigned (SL, Index ID) to that particular sample in the field data sheets.

Samples collected during this investigation will be packaged and shipped IAW CDM SOP 2-1 *Packaging and Shipping of Environmental Samples* (Appendix E) and ASTM Standard D-5755-95 (Appendix B), with modification.

The approved modifications to SOP 2-1 are as follows:

Section 4.0, Required Equipment – No vermiculite or other absorbent material will be used. No bubble wrap or ice will be used.

7.0 Equipment Decontamination

This project requires the decontamination of all personal air sampling, ambient air sampling, and microvacuum dust sampling equipment (e.g., pumps, cassettes, tubing, etc) prior to sampling and prior to leaving the site. Equipment used to collect, handle, or measure samples will be decontaminated.

The decontamination procedure for nondisposable equipment will consist of wet wiping the exposed surfaces. All equipment will then be allowed to air-dry. All equipment will be decontaminated before coming into contact with any sample.

8.0 Quality Assurance/Quality Control

Quality Assurance/Quality Control (QA/QC) requirements will be performed IAW CDM's Quality Implementation Plan for the DOT Volpe National Transportation Center dated June 14, 2000, CDM's Technical Standard Operating Procedures, Revision 15 dated October 12, 2001, and the Sampling and Quality Assurance Project Plan Revision 1 for Libby, Montana, Environmental Monitoring for Asbestos. With the exception of Section B.4, QA/QC requirements contained within the Phase 2 Sampling and Quality Assurance Project Plan Revision 0 for Libby, Montana will also be followed.

9.0 Health and Safety

All sampling will be IAW all applicable EPA, OSHA, corporate, and site health and safety requirements. CDM has prepared a site health and safety plan for the project site that is attached (Appendix G).

Sampling and Analysis Plan Stimson Lumber Company, Libby, Montana Libby Asbestos Project

10.0 References

CDM. 2002. Final Sampling and Analysis Plan for the Remedial Investigation of Contaminant Screening Study. April.

Environmental Protection Agency (EPA). 2000. Sampling and Quality Assurance Project Plan, Revision 1 for Libby, Montana, Environmental Monitoring for Asbestos, Baseline Monitoring for Source Area and Residential Exposure to Tremolite-Actinolite Asbestos Fibers. January.

EPA. 2001. Phase 2 Sampling and Quality Assurance Project Plan Revision 0 for Libby, Montana, Environmental Monitoring for Asbestos, Evaluation of Exposure to Airborne Asbestos Fibers During Routine and Special Activities. 2001. March.

Appendix A

EPA SOP 2015 Asbestos Sampling



ASBESTOS SAMPLING

SOP#: 2015 DATE: 11/17/94 REV. #: 0.0

1.0 SCOPE AND APPLICATION

Asbestos has been used in many commercial products including building materials such as flooring tiles and sheet goods, paints and coatings, insulation, and roofing asphalts. These products and others may be found at hazardous waste sites hanging on overhead pipes, contained in drums, abandoned in piles, or as part of a structure. Asbestos tailing piles from mining operations can also be a source of ambient asbestos fibers. Asbestos is a known carcinogen and requires air sampling to assess airborne exposure to human health. This Standard Operating Procedure (SOP) provides procedures for asbestos air sampling by drawing a known volume of air through a mixed cellulose ester (MCE) filter. The filter is then sent to a laboratory for analysis. The U.S. Environmental Protection Agency/Environmental Response Team (U.S. EPA/ERT) uses one of four analytical methods for determining asbestos in air. These include: U.S. EPA's Environmental Asbestos Assessment Manual. Superfund Method for the Determination of Asbestos in Ambient Air for Transmission Electron Microscopy (TEM)(1); U.S. EPA's Modified Yamate Method for TEM⁽²⁾: National Institute for Occupational Safety and Health (NIOSH) Method 7402 (direct method only) for TEM; and NIOSH Method 7400 for Phase Contrast Microscopy (PCM)(3). Each method has specific sampling and analytical requirements (i.e., sample volume and flow rate) for determining asbestos in air.

The U.S. EPA/ERT typically follows procedures outlined in the TEM methods for determining mineralogical types of asbestos in air and for distinguishing asbestos from non-asbestos minerals. The Phase Contrast Microscopy (PCM) method is used by U.S. EPA/ERT as a screening tool since it is less costly than TEM. PCM cannot distinguish asbestos from non-asbestos fibers, therefore the TEM method may be necessary to confirm analytical results. For example, if an action level for the presence of fibers has been set and PCM analysis indicates that the action level has been exceeded, then

TEM analysis can be used to quantify and identify asbestos structures through examination of their morphology crystal structures (through electron diffraction), and elemental composition (through energy dispersive X-ray analysis). In this instance samples should be collected for both analyses in side by side sampling trains (some laboratories are able to perform PCM and TEM analysis from the same filter). The Superfund method is designed specifically to provide results suitable for supporting risk assessments at Superfund sites, it is applicable to a wide range of ambient air situations at hazardous waste sites. U.S. EPA's Modified Yamate Method for TEM is also used for ambient air sampling due to high volume requirements. The PCM and TEM NIOSH analytical methods require lower sample volumes and are typically used indoors; however, ERT will increase the volume requirement for outdoor application.

Other Regulations pertaining to asbestos have been promulgated by U.S. EPA and OSHA. U.S. EPA's National Emission Standards for Hazardous Air Pollutants (NESHAP) regulates asbestos-containing waste materials. NESHAP establishes management practices and standards for the handling of asbestos and emissions from waste disposal operations (40 CFR Part 61, Subparts A and M). U.S. EPA's 40 CFR 763 (July 1, 1987)⁽⁴⁾ and its addendum 40 CFR 763 (October 30, 1987)⁽⁴⁾ provide comprehensive rules for the asbestos abatement industry. State and local regulations on these issues vary and may be more stringent than federal requirements. The OSHA regulations in 29 CFR 1910.1001 and 29 CFR 1926.58 specify work practices and safety equipment such as respiratory protection and protective clothing when handling asbestos. The OSHA standard for an 8-hour, time-weighted average (TWA) is 0.2 fibers/cubic centimeters of air. This standard pertains to fibers with a length-to-width ratio of 3 to 1 with a fiber length >5 µm^(5.6). An action level of 0.1 fiber/cc (one-half the OSHA standard) is the level U.S. EPA has established in which employers must initiate such activities as air monitoring, employee training, and

medical surveillance(5,6).

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

Prior to sampling, the site should be characterized by identifying on-site as well as off-site sources of-airborne asbestos. The array of sampling locations and the schedule for sample collection, is critical to the success of an investigation. Generally, sampling strategies to characterize a single point source are fairly straightforward, while multiple point sources and area sources increase the complexity of the sampling strategy. It is not within the scope of this SOP to provide a generic asbestos air sampling plan. Experience, objectives, and site characteristics will dictate the sampling strategy.

During a site investigation, sampling stations should be arranged to distinguish spatial trends in airborne asbestos concentrations. Sampling schedules should be fashioned to establish temporal trends. The sampling strategy typically requires that the concentration of asbestos at the source (worst case) or area of concern (downwind), crosswind, as well as background (upwind) contributions be quantified. See Table 1 (Appendix A) for U.S. EPA/ERT recommended sampling set up for ambient air. Indoor asbestos sampling requires a different type of strategy which is identified in Table 2 (Appendix A). It is important to establish background levels of contaminants in order to develop a reference point from which to evaluate the source data. Field blanks and lot blanks can be utilized to determine other sources.

Much information can be derived from each analytical method previously mentioned. Each analytical method has specific sampling requirements and produce results which may or may not be applicable to a specific sampling effort. The site sampling

objectives should be carefully identified so as to select the most appropriate analytical method. Additionally, some preparation (i.e., lot blanks results) prior to site sampling may be required, these requirements are specified in the analytical methods.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

3.1 Sample Preservation

No preservation is required for asbestos samples.

3.2 Sample Handling, Container and Storage Procedures

- 1. Place a sample label on the cassette indicating a unique sampling number. Do not put sampling cassettes in shirt or coat pockets as the filter can pick up fibers. The original cassette box is used to hold the samples.
- 2. Wrap the cassette individually in a plastic sample bag. Each bag should be marked indicating sample identification number, total volume, and date.
- 3. The wrapped sampling cassettes should be placed upright in a rigid container so that the cassette cap is on top and cassette base is on bottom. Use enough packing material to prevent jostling or damage. Do not use vermiculite as packing material for samples. If possible, hand carry to lab.
- 4. Provide appropriate documentation with samples (i.e., chain of custody and requested analytical methodology).

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Flow rates exceeding 16 liters/minute (L/min) which could result in filter destruction due to (a) failure of its physical support under force from the increased pressure drop; (b) leakage of air around the filter mount so that the filter is bypassed, or (c) damage to the asbestos structures due to increased impact velocities.

4.1 U.S. EPA's Superfund Method

4.1.1 Direct-transfer TEM Specimen Preparation Methods

Direct-Transfer TEM specimen preparation methods have the following significant interferences:

- The achievable detection limit is restricted by the particulate density on the filter, which in turn is controlled by the sampled air volume and the total suspended particulate concentration in the atmosphere being sampled.
- The precision of the result is dependent on the uniformity of the deposit of asbestos structures on the sample collection filter.
- Air samples must be collected so that they have particulate and fiber loadings within narrow ranges. If too high a particulate loading occurs on the filter, it is not possible to prepare satisfactory TEM specimens by a direct-transfer method. If too high a fiber loading occurs on the filter, even if satisfactory TEM specimens can be prepared, accurate fiber counting will not be possible.

4.1.2 Indirect TEM Specimen Preparation Methods

Indirect TEM specimen preparation methods have the following interferences:

- The size distribution of asbestos structures is modified.
- There is increased opportunity for fiber loss or introduction of extraneous contamination.
- When sample collection filters are ashed, any fiber contamination in the filter medium is concentrated on the TEM specimen grid.

It can be argued that direct methods yield an underestimate of the asbestos structure concentration because many of the asbestos fibers present are concealed by other particulate material with which they are associated. Conversely, indirect methods can be considered to yield an over-estimate because some types of complex asbestos structures disintegrate during the preparation, resulting in an increase in the numbers of structures counted.

4.2 U.S. EPA's Modified Yamate Method for TEM

High concentrations of background dust interfere with fiber identification.

4.3 NIOSH Method for TEM

Other amphibole particles that have aspect ratios greater than 3:1 and elemental compositions similar to the asbestos minerals may interfere in the TEM analysis. Some non-amphibole minerals may give electron diffraction patterns similar to amphiboles. High concentrations of background dust interfere with fiber identification.

4.4 NIOSH Method for PCM

PCM cannot distinguish asbestos from non-asbestos fibers; therefore, all particles meeting the counting criteria are counted as total asbestos fibers. Fiber less than 0.25 um in length will not be detected by this method. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

5.0 EQUIPMENT/MATERIALS

5.1 Sampling Pump

The constant flow or critical orifice controlled sampling pump should be capable of a flow-rate and pumping time sufficient to achieve the desired volume of air sampled.

The lower flow personal sampling pumps generally provide a flow rate of 20 cubic centimeters/minute (cc/min) to 4 L/min. These pumps are usually battery powered. High flow pumps are utilized when flow rates between 2 L/min to 20 L/min are required. High flow pumps are used for short sampling periods so as to obtain the desired sample volume. High flow pumps usually run on AC power and can be plugged into a nearby outlet. If an outlet is not available then a generator should be obtained. The generator should be positioned downwind from the sampling pump. Additional voltage may be required if more than one pump is plugged into the same generator. Several

electrical extension cords may be required if sampling locations are remote.

The recommended volume for the Superfund method (Phase I) requires approximately 20 hours to collect. Such pumps typically draw 6 amps at full power so that 2 lead/acid batteries should provide sufficient power to collect a full sample. The use of line voltage, where available, eliminates the difficulties associated with transporting stored electrical energy.

A stand should be used to hold the filter cassette at the desired height for sampling and the filter cassette shall be isolated from the vibrations of the pump.

5.2 Filter Cassette

The cassettes are purchased with the required filters in position, or can be assembled in a laminar flow hood or clean area. When the filters are in position, a shrink cellulose band or adhesive tape should be applied to cassette joints to prevent air leakage.

5.2.1 TEM Cassette Requirements

Commercially available field monitors, comprising 25 mm diameter three-piece cassettes, with conductive extension cowls shall be used for sample collection. The cassette must be new and not previously used. The cassette shall be loaded with an MCE filter of pore size 0.45 µm, and supplied from a lot number which has been qualified as low background for asbestos determination. The cowls should be constructed of electrically conducting material to minimize electrostatic effects. The filter shall be backed by a 5 µm pore size MCE filter (Figure 1, Appendix B).

5.2.2 PCM Cassette Requirements

NIOSH Method 7400, PCM involves using a 0.8 to $1.2~\mu m$ mixed cellulose ester membrane, 25 mm diameter, 50 mm conductive cowl on cassette (Figure 2, Appendix B). Some labs are able to perform PCM and TEM analysis on the same filter; however, this should be discussed with the laboratory prior to sampling.

5.3 Other Equipment

- Inert tubing with glass cyclone and hose barb
- Whirlbags (plastic bags) for cassettes

- Tools small screw drivers
- Container to keep samples upright
- Generator or electrical outlet (may not be required)
- Extension cords (may not be required)
 - Multiple plug outlet
- Sample labels
- Air data sheets
- Chain of Custody records

6.0 REAGENTS

Reagents are not required for the preservation of asbestos samples.

7.0 PROCEDURES

7.1 Air Volumes and Flow Rates

Sampling volumes are determined on the basis of how many fibers need to be collected for reliable measurements. Therefore, one must estimate how many airborne fibers may be in the sampling location.

Since the concentration of airborne aerosol contaminants will have some effect on the sample, the following is a suggested criteria to assist in selecting a flow rate based on real-time aerosol monitor (RAM) readings in milligrams/cubic meter (mg/m³).

	Concentratio	n Flow Rate
 Low RAM readings: 	<6.0 mg/m ³	11-15. L/min
 Medium RAM reading 	s:>6.0 mg/m ₃	7.5 L/min
 High RAM readings: 	$>10. \text{ mg/m}^3$	2.5 L/min

In practice, pumps that are available for environmental sampling at remote locations operate under a maximum load of approximately 12 L/min.

7.1.1 U.S. EPA's Superfund Method

The Superfund Method incorporates an indirect preparation procedure to provide flexibility in the amount of deposit that be can be tolerated on the sample filter and to allow for the selective concentration of asbestos prior to analysis. To minimize contributions to background contamination from asbestos present in the plastic matrices of membrane filters while allowing for sufficient quantities of asbestos to be collected, this method also requires the collection of a larger volume of air per unit area of filter than has traditionally been collected

for asbestos analysis. Due to the need to collect large volumes of air, higher sampling flow rates are recommended in this method than have generally been employed for asbestos sampling in the past. As an alternative, samples may be collected over longer time intervals. However, this restricts the flexibility required to allow samples to be collected while uniform meteorological conditions prevail.

The sampling rate and the period of sampling should be selected to yield as high a sampled volume as possible, which will minimize the influence of filter contamination. Wherever possible, a volume of 15 cubic meters (15,000 L) shall be sampled for those samples intended for analysis only by the indirect TEM preparation method (Phase 1 samples). For those samples to be prepared by both the indirect and the direct specimen preparation methods (Phase 2 samples), the volumes must be adjusted so as to provide a suitably-loaded filter for the direct TEM preparation method. One option is to collect filters at several loadings to bracket the estimated optimum loading for a particular site. Such filters can be screened in the laboratory so that only those filters closest to optimal loading are analyzed. It has been found that the volume cannot normally exceed 5 cubic meters (5000 L) in an urban or agricultural area, and 10 cubic meters (10,000 L) in a rural area for samples collected on a 25 mm filter and prepared by a directtransfer technique.

An upper limit to the range of acceptable flow rates for this method is 15 L/min. At many locations, wind patterns exhibit strong diurnal variations. Therefore, intermittent sampling (sampling over a fixed time interval repeated over several days) may be necessary to accumulate 20 hours of sampling time over constant wind conditions. Other sampling objectives also may necessitate intermittent sampling. The objective is to design a sampling schedule so that samples are collected under uniform conditions throughout the sampling interval. This method provides for such options. Air volumes collected on Phase I samples are maximized (<16 L/min). Air volumes collected on Phase 2 samples are limited to provide optimum loading for filters to be prepared by a direct-transfer procedure.

7.1.2 U.S. EPA's Modified Yamate Method for TEM

U.S. EPA's TEM method requires a minimum volume

of 560 L and a maximum volume of 3,800 L in order to obtain an analytical sensitivity of 0.005 structures/cc. The optimal volume for TEM is 1200 L to 1800 L. These volumes are determined using a 200 mesh EM grid opening with a 25-mm filter cassette. Changes in volume would be necessary if a 37-mm filter cassette is used since the effective area of a 25 mm (385 sq mm) and 37 mm (855 sq m) differ.

7.1.3 NIOSH Method for TEM and PCM

The minimum recommended volume for TEM and PCM is 400 L at 0.1 fiber/cc. Sampling time is adjusted to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for eight hours (700 to 2800 L) is appropriate in non-dusty atmospheres containing 0.1 fiber/cc. Dusty atmospheres i.e., areas with high levels of asbestos, require smaller sample volumes (<400 L) to obtain countable samples.

In such cases, take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3,000 to 10,000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If > 50% of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration. Do not exceed 0.5 mg total dust loading on the filter.

7.2 Calibration Procedures

In order to determine if a sampling pump is measuring the flow rate or volume of air correctly, it is necessary to calibrate the instrument. Sampling pumps should be calibrated immediately before and after each use. Preliminary calibration should be conducted using a primary calibrator such as a soap bubble type calibrator, (e.g., a Buck Calibrator, Gilibrator, or equivalent primary calibrator) with a representative filter cassette installed between the pump and the calibrator. The representative sampling cassette can be reused for calibrating other pumps that will be used for asbestos sampling. The same cassette lot used for sampling should also be used for the calibration. A sticker should be affixed to the outside of the extension cowl marked "Calibration Cassette."

A rotameter can be used provided it has been recently precalibrated with a primary calibrator. separate constant flow calibration readings should be obtained both before sampling and after sampling. Should the flow rate change by more than 5% during the sampling period, the average of the pre- and postcalibration rates will be used to calculate the total sample volume. The sampling pump used shall provide a non-fluctuating air-flow through the filter. and shall maintain the initial volume flow-rate to within ± 10% throughout the sampling period. The mean value of these flow-rate measurements shall be used to calculate the total air volume sampled. A constant flow or critical orifice controlled pump meets these requirements. If at any time the measurement indicates that the flow-rate has decreased by more than 30%, the sampling shall be terminated. Flexible tubing is used to connect the filter cassette to thesampling pump. Sampling pumps can be calibrated prior to coming on-site so that time is saved when performing on-site calibration.

- 7.2.1 Calibrating a Personal Sampling Pump with an Electronic Calibrator
- See Manufacturer's manual for operational instructions.
- Set up the calibration train as shown in (Figure 3, Appendix B) using a sampling pump, electronic calibrator, and a representative filter cassette. The same lot sampling cassette used for sampling should also be used for calibrating.
- 3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 foot) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the electronic calibrator.
- 4. Turn the electronic calibrator and sampling pump on. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.
- Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

- Perform the calibration three times until the desired flow rate of ± 5% is attained.
- 7.2.2 Calibrating a Rotameter with an Electronic Calibrator
- See manufacturer's manual for operational instructions.
- Set up the calibration train as shown in (Figure 4, Appendix B) using a sampling pump, rotameter, and electronic calibrator.
- Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6° vertical.
- 4. Turn the electronic calibrator and sampling pump on.
- 5. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.
- Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.
- 7. Record the electronic calibrator flow rate reading and the corresponding rotameter reading. Indicate these values on the rotameter (sticker). The rotameter should be able to work within the desired flow range. Readings can also be calibrated for 10 cm³ increments for Low Flow rotameters, 500 cm³ increments for medium flow rotameters and 1 liter increments for high flow rotameters.
- 8. Perform the calibration three times until the desired flow rate of \pm 5% is attained. Once on site, a secondary calibrator, i.e., rotameter may be used to calibrate sampling pumps.
- 7.2.3 Calibrating a Personal Sampling
 Pump with a Rotameter
- See manufacturer's manual for Rotameter's Operational Instructions.

- 2. Set up the calibration train as shown in (Figure 5, Appendix B) using a rotameter, sampling pump, and a representative sampling cassette.
- To set up the calibration train, attach one end
 of the PVC tubing (approx. 2 ft) to the
 cassette base; attach the other end of the
 tubing to the inlet plug on the pump.
 Another piece of tubing is attached from the
 cassette cap to the rotameter.
- Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6° vertical.
- 5. Turn the sampling pump on.
- Turn the flow adjust screw (or knob) on the
 personal sampling pump until the float ball
 on the rotameter is lined up with the
 precalibrated flow rate value. A sticker on
 the rotameter should indicate this value.
- A verification of calibration is generally performed on-site in the clean zone immediately prior to the sampling.

7.3. Meteorology

It is recommended that a meteorological station be established. If possible, sample after two to three days of dry weather and when the wind conditions are at 10 mph or greater. Record wind speed, wind direction, temperature, and pressure in a field logbook. Wind 'direction is particularly important when monitoring for asbestos downwind from a fixed source.

7.4 Ambient Sampling Procedures

7.4.1 Pre-site Sampling Preparation

- Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.
- Obtain necessary sampling equipment and ensure it is in working order and fully charged (if necessary).

- 3. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety plan.
- Once on-site the calibration is performed in the clean zone. The calibration procedures are listed in Section 7.2.
- 5. After calibrating the sampling pump, mobilize to the sampling location.

7.4.2 Site Sampling

- 1. To set up the sampling train, attach the air intake hose to the cassette base. Remove the cassette cap (Figure 6 and 7, Appendix B). The cassette should be positioned downward, perpendicular to the wind
- If AC or DC electricity is required then turn it on. If used, the generator should be placed 10 ft. downwind from the sampling pump.
- Record the following in a field logbook: date, time, location, sample identification number, pump number, flow rate, and cumulative time.
- 4. Turn the pump on. Should intermittent sampling be required, sampling filters must be covered between active periods of sampling. To cover the sample filter: turn the cassette to face upward, place the cassette cap on the cassette, remove the inlet plug from the cassette cap, attach a rotameter to the inlet opening of the cassette cap to measure the flow rate, turn off the sampling pump, place the inlet plug into the inlet opening on the cassette cap. To resume sampling: remove the inlet plug, turn on the sampling pump, attach a rotameter to measure the flow rate, remove the cassette cap, replace the inlet plug in the cassette cap and invert the cassette, face downward and perpendicular to the wind.
- Check the pump at sampling midpoint if sampling is longer than 4 hours. The generators may need to be regased depending on tank size. If a filter darkens in appearance or if loose dust is seen in the filter, a second sample should be started.

- 6. At the end of the sampling period, orient the cassette up, turn the pump off.
- 7. Check the flow rate as shown in Section 7.2.3. When sampling open-faced, the sampling cap should be replaced before post calibrating. Use the same cassette used for sampling for post calibration (increase dust/fiber loading may have altered the flow rate.
- 8. Record the post flow rate.
- 9. Record the cumulative time or run.
- 10. Remove the tubing from the sampling cassette. Still holding the cassette upright, replace the inlet plug on the cassette cap and the outlet plug on the cassette base.

7.4.3. Post Site Sampling

- Follow handling procedures in Section 3.2, steps 1-4.
- Obtain an electronic or hard copy of meteorological data which occurred during the sampling event. Record weather: wind speed, ambient temperature, wind direction, and precipitation. Obtaining weather data several days prior to the sampling event can also be useful.

7.5 Indoor Sampling Procedures

PCM analysis is used for indoor air samples. When analysis shows total fiber count above the OSHA action level 0.1 f/cc then TEM (U.S. EPA's Modified Yamate Method) is used to identify asbestos from non-asbestos fibers.

Sampling pumps should be placed four to five feet above ground level away from obstructions that may influence air flow. The pump can be placed on a table or counter. Refer to Table 2 (Appendix A) for a summary of indoor sampling locations and rationale for selection.

Indoor sampling utilizes high flow rates to increased sample volumes (2000 L for PCM and 2800 to 4200 L for TEM) in order to obtain lower detection limits below the standard, (i.e., 0.01 f/cc or lower [PCM]

and 0.005 structures/cc or lower [TEM]).

7.5.1 Aggressive Sampling Procedures

Sampling equipment at fixed locations may fail to detect the presence of asbestos fibers. Due to limited air movement, many fibers may settle out of the air onto the floor and other surfaces and may not be captured on the filter. In the past, an 8-hour sampling period was recommended to cover various air circulation conditions. A quicker and more effective way to capture asbestos fibers is to circulate the air artificially so that the fibers remain airborne during sampling. The results from this sampling option typifies worst case condition. This is referred to as aggressive air sampling for asbestos. Refer to Table 2 for sample station locations.

- 1. Before starting the sampling pumps, direct forced air (such as a 1-horsepower leaf blower or large fan) against walls, ceilings, floors, ledges, and other surfaces in the room to initially dislodge fibers from surfaces. This should take at least 5 minutes per 1000 sq. ft. of floor.
- Place a 20-inch fan in the center of the room.
 (Use one fan per 10,000 cubic feet of room space.) Place the fan on slow speed and point it toward the ceiling.
- 3. Follow procedures in Section 7.4.1 and 7.4.2 (Turn off the pump and then the fan(s) when sampling is complete.).
- 4. Follow handling procedures in Section 3.2, steps 1-4.

8.0 CALCULATIONS

The sample volume is calculated from the average flow rate of the pump multiplied by the number of minutes the pump was running (volume = flow rate X time in minutes). The sample volume should be submitted to the laboratory and identified on the chain of custody for each sample (zero for lot, field and trip blanks).

The concentration result is calculated using the sample volume and the numbers of asbestos structures reported after the application of the cluster and matrix counting criteria.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

Follow all QA/QC requirements from the laboratories as well as the analytical methods.

9.1 TEM Requirements

- 1. Examine lot blanks to determine the background asbestos structure concentration.
- 2. Examine field blanks to determine whether there is contamination by extraneous asbestos structures during specimen preparation.
- 3. Examine of laboratory blanks to determine if contamination is being introduced during critical phases of the laboratory program.
- 4. To determine if the laboratory can satisfactorily analyze samples of known asbestos structure concentrations, reference filters shall be examined. Reference filters should be maintained as part of the laboratory's Quality Assurance program.
- 5. To minimize subjective effects, some specimens should be recounted by a different microscopist.
- Asbestos laboratories shall be accredited by the National Voluntary Laboratory Accreditation Program.
- At this time, performance evaluation samples for asbestos in air are not available for Removal Program Activities.

9.2 PCM Requirements

- Examine reference slides of known concentration to determine the analyst's ability to satisfactorily count fibers. Reference slides should be maintained as part of the laboratory's quality assurance program.
- Examine field blanks to determine if there is contamination by extraneous structures during sample handling.

- Some samples should be relabeled then submitted for counting by the same analyst to determine possible bias by the analyst.
- 4. Participation in a proficiency testing program such as the AIHA-NIOSH proficiency analytical testing (PAT) program.

10.0 DATA VALIDATION

Results of quality control samples will be evaluated for contamination. This information will be utilized to qualify the environmental sample results accordingly with the project's data quality objectives.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety procedures. More specifically, when entering an unknown situation involving asbestos, a powered air purifying respirator (PAPR) (full face-piece) is necessary in conjunction with HEPA filter cartridges. See applicable regulations for action level, PEL, TLV, etc. If previous sampling indicates asbestos concentrations are below personal health and safety levels, then Level D personal protection is adequate.

12.0 REFERENCES

- Environmental Asbestos Assessment Manual, Superfund Method for the Determination of Asbestos in Ambient Air, Part 1: Method, EPA/540/2-90/005a, May 1990, and Part 2: Technical Background Document, EPA/540/2-90/005b, May 1990.
- Methodology for the Measurement of Airborne Asbestos by Electron Microscopy, EPA's Report No. 68-02-3266, 1984, G. Yamate, S.C. Agarwal, and R. D. Gibbons.
- National Institute for Occupational Safety and Health. NIOSH Manual of Analytical Method. Third Edition. 1987.
- U.S. Environmental Protection Agency. Code of Federal Regulations 40 CFR 763. July 1, 1987. Code of Federal Regulations 40 CFR 763 Addendum. October 30, 1987.

- U.S. Environmental Protection Agency.

 Asbestos-Containing Materials in Schools;
 Final Rule and Notice. 52 FR 41826.
- Occupational Safety and Health Administration. Code of Federal Regulations 29 CFR 1910.1001. Washington, D.C. 1987.

(6)

APPENDIX A

Tables

TABLE 1. SAMPLE STATIONS FOR OUTDOOR SAMPLING			
Sample Station Location	Sample Numbers -	Rationale	
Upwind/Background ⁽¹⁾	Collect a minimum of two simultaneous upwind/background samples 30° apart from the prevailing windlines.	Establishes background fiber levels.	
Downwind	Deploy a minimum of 3 sampling stations in a 180 degree arc downwind from the source.	Indicates if asbestos is leaving the site.	
Site Representative and/or Worst Case	Obtain one site representative sample which shows average condition on-site or obtain worst case sample (optional).	Verify and continually confirm and document selection of proper levels of worker protection.	

⁽¹⁾ More than one background station may be required if the asbestos originates from different sources.

Tables

TABLE 2 SAMPLE STATIONS FOR INDOOR SAMPLING					
Sample Station Location	Sample Numbers	Rationale			
Indoor Sampling	If a work site is a single room, disperse 5 samplers throughout the room. If the work site contains up to 5 rooms, place at least one sampler in each room. If the work site contains more than 5 rooms, select a representative sample of the rooms.	Establishes representative samples from a homogeneous area.			
Upwind/Background	If outside sources are suspected, deploy a minimum of two simultaneous upwind/background samples 30° apart from the prevailing windlines.	Establish whether indoor asbestos concentrations are coming from an outside source.			
Worst Case	Obtain one worst case sample, i.e., aggressive sampling (optional).	Verify and continually confirm and document selection of proper levels of worker protection.			

APPENDIX B

FIGURE 1. Transmission Electron Microscopy Filter Cassette

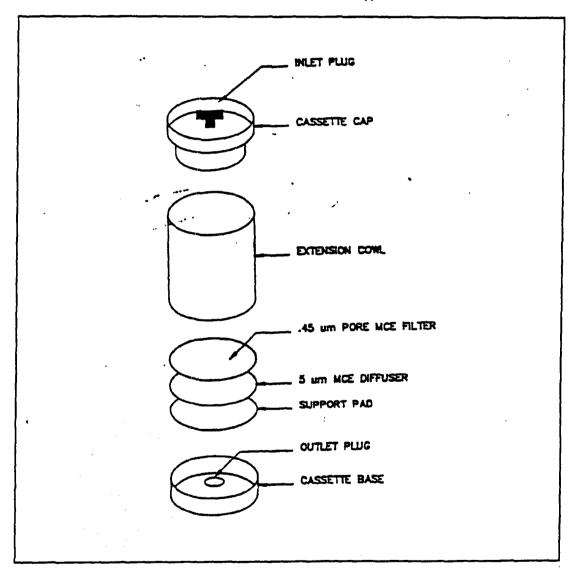


FIGURE 2. Phase Contrast Microscopy Filter Cassette

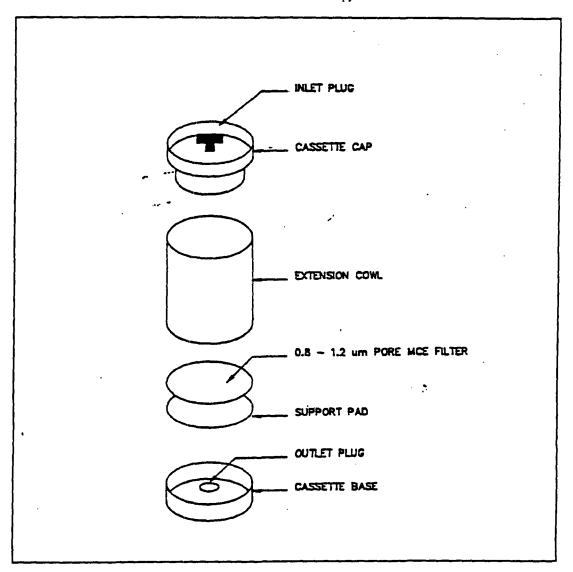


FIGURE 3. Calibrating a Personal Sampling Pump with a Bubble Meter

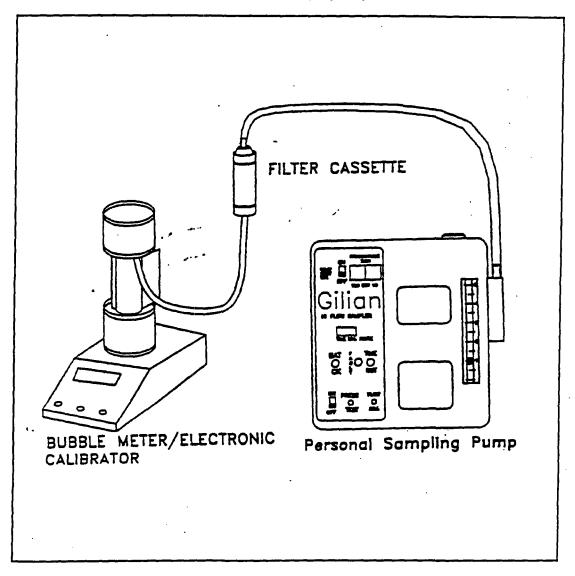


FIGURE 4. Calibrating a Rotameter with a Bubble Meter

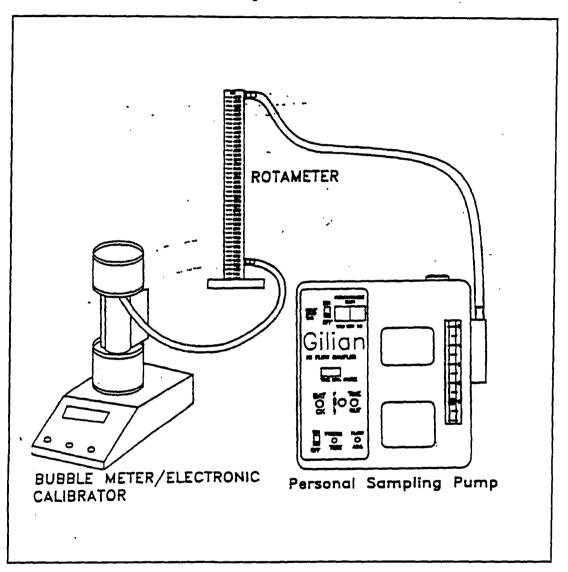


FIGURE 5. Calibrating a Sampling Pump with a Rotameter

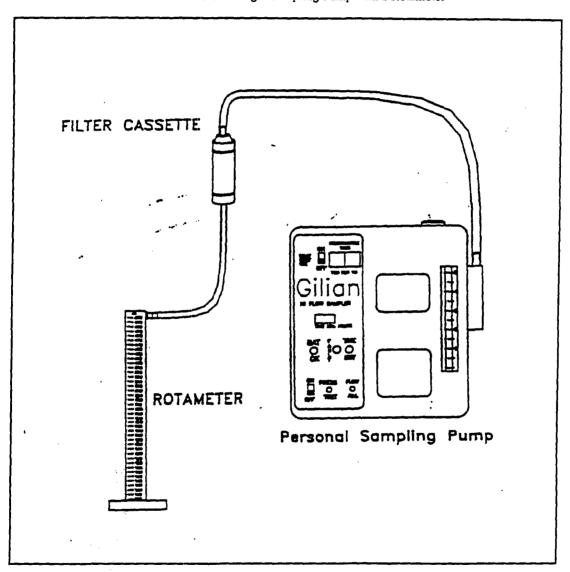


FIGURE 6. Personal Sampling Train for Asbestos

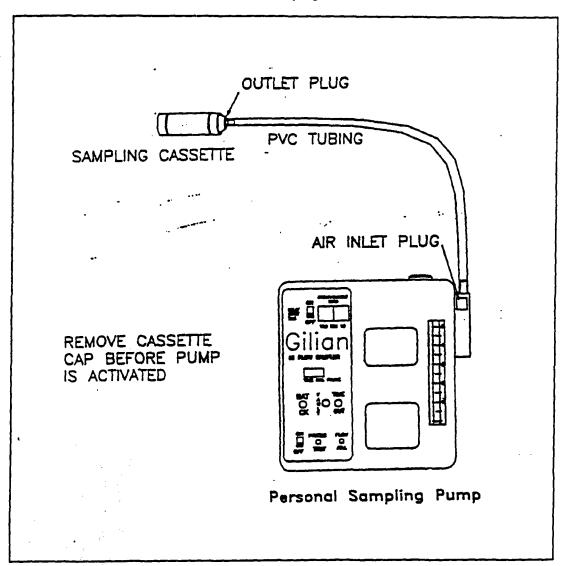
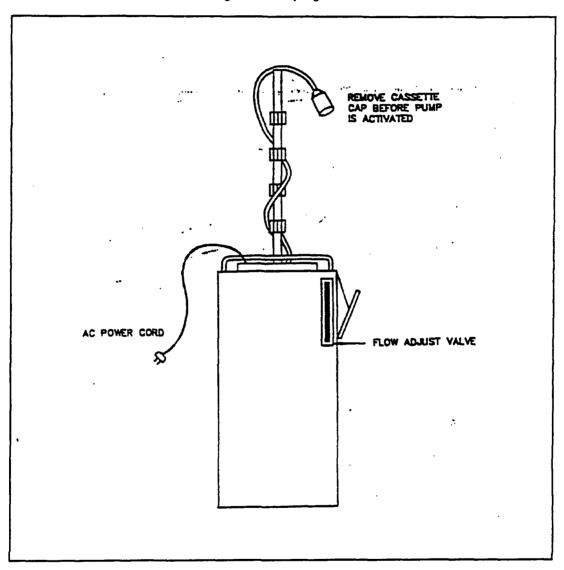


FIGURE 7. High Flow Sampling Train for Asbestos



Appendix B

ASTM Standard 5755-95
Standard Test Method for Microvacuum Sampling and Indirect
Analysis of Dust by Transmission Electron Microscopy for Asbestos
Structure Number Concentrations



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if not listed in the current contributed index, well appear in the next estion.

Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations¹

This standard is issued under the fixed designation D 1755; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last responsel, A superscript epsilon (c) indicates an editorial change since the last revision or reapproval.

1. Scope

- 1.1 This test method covers a procedure to (a) identify asbestos in dust and (b) provide an estimate of the concentration of asbestos in the sampled dust reported as the number of asbestos structures per unit area of sampled surface.
- 1.1.1 If an estimate of the asbestos mass is to be determined, the user is referred to Test Method D 5756.
- 1.2 This test method describes the equipment and procedures necessary for sampling, by a microvacuum technique, non-airborne dust for levels of asbestos structures. The non-airborne sample is collected inside a standard filter membrane cassette from the sampling of a surface area for dust which may contain asbestos.
- 1.2.1 This procedure uses a microvacuuming sampling technique. The collection efficiency of this technique is unknown and will vary among substrates. Properties influencing collection efficiency include surface texture, adhesiveness, electrostatic properties and other factors.
- 1.3 Asbestos identified by transmission electron microscopy (TEM) is based on morphology, selected area electron diffraction (SAED), and energy dispersive X-ray analysis (EDXA). Some information about structure size is also determined.
- 1.4 This test method is generally applicable for an estimate of the concentration of asbestos structures starting from approximately 1000 asbestos structures per square centimetre.
- 1.4.1 The procedure outlined in this test method employs an indirect sample preparation technique. It is intended to disperse aggregated asbestos into fundamental fibrils, fiber bundles, clusters, or matrices that can be more accurately quantified by transmission electron microscopy. However, as with all indirect sample preparation techniques, the asbestos observed for quantification may not represent the physical form of the asbestos as sampled. More specifically, the procedure described neither creates nor destroys asbestos, but it may after the physical form of the mineral fibers.
- 1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.
- 1.6 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the

Sampting and Analysis of Atmospheres and is the direct responsibility of

responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

- D 1739 Test Method for the Collection and Measurement of Dustfall (Settleable Particulate Matter)
- D3195 Practice for Rotameter Calibration³
- D 3670 Guide for Determination of Precision and Bias of Methods of Committee D-223
- D 5756 Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Concentration³

3. Terminology

- 3.1 Definitions:
- 3.1.1 asbestiform—a special type of fibrous habit in which the fibers are separable into thinner fibers and ultimately into fibrils. This habit accounts for greater flexibility and higher tensile strength than other habits of the same mineral. For more information on asbestiform mineralogy, see Refs (1), 4 (2) and (3).
- 3.1.2 asbestos—a collective term that describes a group of naturally occurring, inorganic, highly fibrous, silicate dominated minerals, which are easily separated into long, thin, flexible fibers when crushed or processed.

Discussion—Included in the definition are the asbestiform varieties of: serpentine (chrysotile); rebeckite (crocidolite); grunerite (grunerite asbestos); anthophyllite (anthophyllite asbestos); tremolite (tremolite asbestos); and actinolite (actinolite asbestos). The amphibole mineral compositions are defined according to nomenclature of the International Mineralogical Association (3).

Asbestos	Chemical Abstract Service No.3	
Chrysotile	12001-29-5	
Crocidolite	12001-18-4	
Grunerite Asbestos	12172-73-5	
Anthophyllite Asbestos	77536-67-5	
Tremoiite Asbestos	77536-68-6	
Actinolite Asbettos	77536-66-4	

3.1.3 fibril—a single fiber that cannot be separated into

^{*}This test method is under the jurisdiction of ASTM Committee D-22 on

Subcommittee D22.07 on Sampling and Analysis of Asbestos.
Current edition approved August 15, 1995. Published October 1995.

² Annual Book of ASTM Standards, Vol 11.01. ³ Annual Book of ASTM Standards, Vol 11.03.

^{*} The boldface numbers in parentheues refer to the list of references at the end of this test method.

³The non-exhestiferm variations of the minerals indicated in 5.1.3 have different Chemical Abstract Service (CAS) numbers.

smaller components without losing its fibrous properties or appearance.

- 3.2 Descriptions of Terms Specific to This Standard:
- 3.2.1 aspect ratio—the ratio of the length of a fibrous particle to its average width.
- 3.2.2 bundle—a structure composed of three or more fibers in a parallel arrangement with the fibers closer than one fiber diameter to each other.
- 3.2.3 chuster—a structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings of fibers must have more than two points touching.
- 3.2.4 debris—materials that are of an amount and size (particles greater than 1 mm in diameter) that can be visually identified as to their source.
- 3.2.5 dust—any material composed of particles in a size range of ≤1 mm and large enough to settle by virtue of their weight from the ambient air (see definition for settleable particulate matter in Test Method D 1739).
- 3.2.6 fiber—a structure having a minimum length of 0.5 µm, an aspect ratio of 5:1 or greater, and substantially parallel sides (4).
- 3.2.7 fibrous—of a mineral composed of parallel, radiating, or interlaced aggregates of fibers, from which the fibers are sometimes separable. That is, the crystalline aggregate may be referred to as fibrous even if it is not composed of separable fibers, but has that distinct appearance. The term fibrous is used in a general mineralogical way to describe aggregates of grains that crystallize in a needle-like habit and appear to be composed of fibers. Fibrous has a much more general meaning than aspessos. While it is correct that all aspessos minerals are fibrous, not all minerals having fibrous habits are aspessos.
- 3.2.8 indirect preparation—a method in which a sample passes through one or more intermediate steps prior to final filtration.
- 3.2.9 matrix—a structure in which one or more fibers, or fiber bundles that are touching, are attached to, or partially concealed by a single particle or connected group of non-fibrous particles. The exposed fiber must meet the fiber definition (see 3.2.6).
- 3.2.10 structures—a term that is used to categorize all the types of asbestos particles which are recorded during the analysis (such as fibers, bundles, clusters, and matrices). Final results of the test are always expressed in asbestos structures per square continuetre.

4. Summery of Test Method

4.1 The sample is collected by vacuuming a known surface area with a standard 25 or 37 mm air sampling cassette using a plastic tube that is attached to the inlet orifice which acts as a nozzle. The sample is transferred from inside the cassette to an aqueous solution of known volume. Aliquots of the suspension are then filtered through a membrane. A section of the membrane is prepared and transferred to a TEM grid using the direct transfer method. The asbestiform structures are identified, sized, and counted by TEM, using SAED and EDXA at a magnification of 15 000 to 20 000X.

5. Significance and Use

- 5.1 This microvacuum sampling and indirect analysis method is used for the general testing of non-airborne dust samples for asbestos. It is used to assist in the evaluation of dust that may be found on surfaces in buildings such as ceiling tiles, shelving, electrical components, duct work, carpet, etc. This test method provides an index of the concentration of asbestos structures in the dust per unit area analyzed as derived from a quantitative TEM analysis.
- 5.1.1 This test method does not describe procedures or techniques required to evaluate the safety or habitability of buildings with asbestos-containing materials, or compliance with federal, state, or local regulations or statutes. It is the user's responsibility to make these determinations.
- 5.1.2 At present, a single direct relationship between asbestos-containing dust and potential human exposure does not exist. Accordingly, the user should consider these data in relationship to other available information in their evaluation.
- 5.2 This test method uses the definition, settlesble particulate material, found in Test Method D 1739 as the definition of dust. This definition accepts all particles small enough to pass through a 1 mm (No. 18) screen. Thus, a single, large asbestos containing particle(s) (from the large end of the particle size distribution) dispersed during sample preparation may result in anomalously large asbestos concentration results in the TEM analyses of that sample. It is, therefore, recommended that multiple independent samples are secured from the same area, and a minimum of three samples analyzed by the entire procedure.

6. Interferences

- 6.1 The following minerals have properties (that is, chemical or crystalline structure) which are very similar to asbestos minerals and may interfere with the analysis by causing a false positive to be recorded during the test. Therefore, literature references for these materials must be maintained in the laboratory for comparison to asbestos minerals so that they are not misidentified as asbestos minerals.
- 6.1.1 Antigorite.
- 6.1.2 Palygorskite (Attapulgite).
- 6.1.3 Halloysite.
- 6.1.4 Pyroxenes.
- 6.1.5 Sevialite.
- 6.1.6 Vermiculite scrolls.
- 6.1.7 Fibrous taic.
- 6.1.8 Hornblende and other amphiboles other than those listed in 3.1.2.
- 6.2 Collecting any dust particles greater than 1 mm in size in this test method may cause an interference and, therefore, must be avoided.

7. Materials and Equipment

7.1 Purity of Rengents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without

lessening the accuracy of the determination.4

1.2 Transmission Electron Microscope (TEM), an 80 to 120 kV TEM, capable of performing electron diffraction, with a fluorescent screen inscribed with calibrated gradations, is required. The TEM must be equipped with energy dispersive X-ray spectroscopy (EDXA) and it must have a seanning transmission electron microscopy (STEM) attachment or be capable of producing a spot size of less than 250 nm in diameter in crossover.

1.3 Energy Dispersive X-ray System (EDXA).

7.4 High Vacuum Carbon Evaporator, with rotating stage.

15 High Efficiency Particulate Air (HEPA), filtered negative flow hood.

7.5 Exhaust or Fume Hood.

7.7 Particle-free Water (ASTM Type II, see Specification D 1193).

7.8 Glass Beakers (50 mL).

7.9 Glass Sample Containers, with wide mouth screw cap (200 mL) or equivalent sealable container (height of the glass sample container should be approximately 13 cm high by 6 cm wide).

7.10 Waterproof Markers.

7.11 Forceps (tweezers).

7.12 Ultrasonic Bath, table top model (100 W).

7.13 Graduated Pipettes (1, 5, 10 mL sizes), glass or plastic.

7.14 Filter Funnel, either 25 mm or 47 mm, glass or disposable. Filter funnel assemblies, either glass or disposable plastic, and using either a 25 mm or 47 mm diameter filter.

7.15 Side Arm Filter Flask, 1000 mL.

7.16 Mixed Cellulose Exter (MCE) Membrane Filters, 25 or 47 mm diameter, ≤0.22 µm and 5 µm pore size.

7.17 Polycarbonate (PC) Filters, 25 or 47 mm diameter, ≤0.2 µm pore size.

7.18 Storage Containers, for the 25 or 47 mm filters (for archiving).

7.19 Glass Slides, approximately 76 by 25 mm in size.

7.20 Scalpel Blades, No. 10, or equivalent.

7.21 Cabinet-type Desiccator, or low temperature drying oven.

7.22 Chloroform, reagent grade.

7.23 Acetone, reagent grade.

7.24 Dimethylformamide (DMF).

7.25 Glacial Acetic Acid.

7.26 I-methyl-2-pyrrolidone.

7.27 Plasma Asher, low temperature.

7.28 pH Paper.

7.29 Air Sampling Pump, low volume personal-type, capable of achieving a flow rate of 1 to 5 L/min.

7.30 Rotameter.

7.31 Air Sampling Cassettes, 25 mm or 37 mm, containing 0.8 µm or smaller pore size MCE or PC filters.

7.32 Cork Borer, 7 mm.

7.33 Nan-Asbestos Mineral, references as outlined in 6.1.

7.34 Asbestos Standards, as outlined in 3.1.2.

7.35 Tygon Tubing, or equivalent.

7.36 Small Vacuum Pump, that can maintain a pressure of 92 kPa.

7.37 Petri Dishes, large glass, approximately 90 mm in diameter.

7.38 Jaffe Washer, stainless steel or aluminum mesh screen, 30 to 40 mesh, and approximately 75 mm by 50 mm in size.

7.39 Copper TEM Finder Grids, 200 mesh.

7.40 Carbon Evaporator Rods.

7.41 Lens Tissue.

7.42 Ashless Filter Paper Filters, 90 mm diameter,

7.43 Gummed Paper Reinforcement Rings.

7.44 Wash Bottles, plastic.

7.45 Reagent Alcohol, HPLC Grade (Fisher A995 or equivalent).

7.46 Opening Mesh Screen, plastic, 1.0 by 1.0 mm, (Spectra-Mesh #146410 or equivalent).

7.47 Diffraction Grating Replica.

8. Sampling Procedure for Microvacuum Technique

8.1 For sampling asbestos-containing dust in either indoor or outdoor environments, commercially available cassettes must be used. Air monitoring cassettes containing 25 mm or 37 mm diameter mixed cellulose ester (MCE) or polycarbonate (PC) filter membranes with a pore size less than or equal to 0.8 µm are required (7.31). The number of samples collected depends upon the specific circumstances of the study.

8.2 Maintain a log of all pertinent sampling information and sampling locations.

8.3 Sampling pumps and flow indicators shall be catibrated using a certified standard apparatus or assembly (see Practice D 3195 and 7.29).

8.4 Record all calibration information (5).

8.5 Perform a leak check of the sampling system at each sampling site by activating the pump (7.29) with the closed sampling cassette in line. Any air flow shows that a leak is present that must be eliminated before initiating the sampling operation.

8.6 Attach the sampling cassette to the sampling pump at the outlet side of the cassette with plastic tubing (7.35). The plastic tubing must be long enough in that the sample areas can be reached without interference from the sampling pump. Attach a clean, approximately 25.4 mm long piece of plastic tubing (6.35 mm internal diameter) directly to the inlet orifice. Use this piece of tubing as the sampling nozzle. Cut the sampling end of the tubing at a 45° angle as illustrated in Fig. 1. The exact design of the nozzle is not critical as long as some vacuum break is provided to avoid simply pushing the dust around on the surface with the nozzle rather than vacuuming it into the cassette. The internal diameter of the nozzle and flow rate of the pump may vary as long as the air velocity is $100 (\pm 10)$ cm/s. This air velocity calculation is based on an internal sampling tube diameter of 6.35 mm at a flow rate of 2 L/min.

8.7 Measure and determine the sample area of interest. A

^{*} Rusquest Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC, For suggestions on the testing of resignations for the testing of resignations for the American Chemical Society, see Anolar Standards for Luboratory Chemicals, BDH Ltd., Poole, Dorset, U.X., and the United States Pharmacopena and National Formulary, U.S. Pharmacouncal Convention, Inc. (USPC), Rockville, MD.

^{*}Tygon is a registered undemark of the DuPont Co.

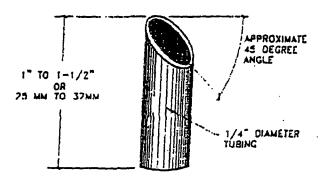


FIG. 1 Example of the Tubing Nozzle

sample area of 100 cm² is vacuumed until there is no visible dust or particulates matter remaining. Perform a minimum of two orthogonal passes on the surface within a minimum of 2 min of sampling time. Avoid scraping or abrading the surface being sampled. (Do not sample any debris or dust particles greater than 1 mm in diameter (see 4.2).) Smaller or larger areas can be sampled, if needed. For example, some surfaces of interest may have a smaller area than 100 cm². Less dusty surfaces may require vacuuming of larger areas. Unlike air samples, the overloading of the cassettes with dust will not be a problem. As defined in 3.2.5, only dust shall be collected for this analysis.

8.8 At the end of sample collection, invert the cassette so that the nozzle inlet faces up before shutting off the power to the pump. The nozzle is then sealed with a cassette end-plug and the cassette/nozzle taped or appropriately packaged to prevent separation of the nozzle and cassette assembly. A second option is the removal of the nozzle from the cassette, then plugging of the cassette and shipment of the nozzle (also plugged at both ends) sealed in a separate closeable plastic bag. A third option is placing the nozzle inside the cassette for shipment. The nozzle is always saved and rinsed because a significant percentage of the dust drawn from a lightly loaded surface may adhere to the inside walls of the tubing.

8.9 Check that all samples are clearly labeled, that all dust sampling information sheets are completed, and that all-pertinent information has been enclosed, in accordance with laboratory quality control practices, before transfer of the samples to the laboratory. Include an unused cassette and nozzle as a field blank.

8.10 Wipe off the exterior surface of the cassettes with disposable wet towels (baby wipes) prior to packaging for shipment.

9. Sample Shipment

9.1 Ship dust samples to an analytical laboratory in a sealed container, but separate from any bulk or air samples. The cassettes must be tightly sealed and packed in a material free of fibers or dust to minimize the potential for contamination. Plastic "bubble pack" is probably the most appropriate material for this purpose.

10. Sample Preparation

10.1 Under a negative flow HEPA hood (7.5), carefully wet-wipe the exterior of the cassettes to remove any possible

contamination before taking cassettes into a clean preparation area.

10.2 Perform sample preparation in a clean facility that has a separate work area from both the bulk and air sample preparation areas.

10.3 Initial specimen preparation shall take place in a clean HEPA filtered negative pressure hood to avoid any possible contamination of the laboratory or personnel, or both, by the potentially large number of asbestos structures in an asbestos-containing dust sample. Cleanliness of the preparation area hoods is measured by the cumulative process blank concentrations (see Section 11).

10.4 All sample preparation steps 10.4.1 through 10.4.6 shall take place in the dust preparation area inside a HEPA hood.

10.4.1 Remove the upper plug from the sample cassette and carefully introduce approximately 10 mL solution of a 50/50 mixture of particle-free water and reagent alcohol into the cassette using a plastic wash bottle (7.44). If the plugged nozzle was left attached to the cassette, then remove the plug and introduce the water/alcohol solution into the cassette through the tubing, and then remove the tubing, if it is visibly clean.

10.4.2 Replace the upper plug or the sample cap and lightly shake the dust suspension by hand for 3 s.

10.4.3 Remove the entire cap of the cassette and pour the suspension through a 1.0 by 1.0 mm opening screen (7.46) into a pre-cleaned 200 mL glass specimen bottle (7.9). All visible traces of the sample contained in the cassette shall be rinsed through the screen into the specimen bottle with a plastic wash bottle containing the 50/50 solution of particle-free water and alcohol. Repeat this procedure two additional times for a total of three washings. Next, rinse the nozzle two or three times through the screen into the specimen bottle with the 50/50 mixture of water and alcohol. Typically, the total amount of the 50/50 mixture used in the rinse is 50 to 75 mL. Discard the 1.0 by 1.0 mm screen and bring the volume of solution in the specimen bottle up to the 100 mL mark on the side of the bottle with particle-free water only.

10.4.4 Adjust the pH of the suspension to 3 to 4 using a 10.0 % solution of acetic acid. Use pH paper for testing. Filter the suspension within 24 h to avoid problems associated with bacterial and fungal growth.

10.4.5 Use either a disposable plastic filtration unit or a glass filtering unit (7.14) for filtration of aliquots of the suspension. The ability of an individual filtration unit to produce a uniform distribution may be tested by the filtration of a colored particulate suspension such as diluted India ink (suspension of carbon black).

10.4.5.1 If a disposable plastic filter funnel unit is used, then unwrap a new disposable plastic filter funnel unit (either 25 or 47 mm diameter) and remove the tape around the base of the funnel. Remove the funnel and discard the top filter supplied with the apparatus, retaining the coarse polypropylene support pad in place. Assemble the unit with the adapter and a properly sized neoprene stopper, and attach the funnel to the 1000 mL side-arm vacuum flask (7.15). Place a 5.0 µm pore size MCE (backing filter) on the support pad. Wet it with a few mL of particle-free water and place an MCE (7.16) or PC filter (≤0.22 µm pore size) (7.17) on top of the backing filter. Apply a vacuum (7.36), ensuring

that the filters are centered and pulled flat without air bubbles. Any irregularities on the filter surface requires the discard of that filter. After the filter has been seated properly, replace the funnel and reseal it with the tape. Return the flask to atmospheric pressure.

10.4.5.2 If a glass filtration unit is used, place a 5 µm pore size MCE (backing filter) on the glass frit surface. Wet the filter with particle-free water, and place an MCE or PC filter (≤0.22 µm pore size) on top of the backing lilter. Apply a vacuum, ensuring that the filters are centered and pulled flat without air bubbles. Replace the filters if any irregularities are seen on the filter surface. Before filtration of each set of sample aliquots, prepare a blank filter by filtration of 50 mL of particle-free water. If aliquots of the same sample are filtered in order of increasing concentration, the glass filtration unit need not be washed between filtration. After completion of the filtration, do not allow the filtration funnel assembly to dry because contamination is then more difficult to remove. Wash any residual suspension from the filtration. assembly by holding it under a flow of water, then rub the surface with a clean paper towel soaked in a detergent solution. Repeat the cleaning operation, and then rinse two times in particle-free water.

10.4.6 With the flask at atmospheric pressure, add 20 mL of particle-free water into the funnel. Cover the filter funnel with its plastic cover if the disposable filtering unit is used.

10.4.7 Briefly hand shake (3 s) the capped bottle with the sample suspension, then place it in a tabletop ultrasonic bath (7.12) and sonicate for 3.0 min. Maintain the water level in the sonicator at the same height as the solution in sample bottle. The ultrasonic bath shall be calibrated as described in 20.5. The ultrasonic bath must be operated at equilibrium temperature. After sonicating, return the sample bottle to the work surface of the HEPA hood. Preparation steps 10.4.8 through 10.4.14 shall be carried out in this hood.

10.4.8 Shake the suspension lightly by hand for 3 s, then let it rest for 2.0 min to allow large particles to settle to the bottom of the bottle or float to the surface.

10.4.9 Estimate the amount of liquid to be withdrawn to produce an adequate filter preparation. Experience has shown that a light staining of the filter surface will yield a suitable preparation for analysis. Filter at least 1.0 mL, but no more than half the total volume. If after examination in the TEM, the smallest volume measured (1.0 mL) (7.13) yields an overloaded sample, then perform additional serial dilutions of the suspension. If it is estimated that less than 1.0 mL of solution has to be filtered because of the density of the suspension, perform a serial dilution.

10.4.9.1 If serial dilutions are required, repeat step 10.4.8 before the serial dilution portion is taken. Do not re-sonicate the original solution or any serial dilutions. The recommended procedure for a serial dilution is to mix 10 mL of the sample solution with 90 mL of particle-free water in a clean sample bottle to obtain a 1:10 serial dilution. Follow good laboratory practices when performing dilutions.

10.4.10 Insert a new disposable pipette halfway into the sample suspension and withdraw a portion. Avoid pipetting any of the large floating or settled particles. Uncover the filter funnel and dispense the mixture from the pipette into the water in the funnel.

10.4.11 Apply vacuum to the flask and draw the mixture through the filter.

10.4.12 Discard the pipette.

10.4.13 Disassemble the filtering unit and carefully remove the sample filter with fine tweezers (7.11). Place the completed sample filter particle side up, into a precleaned, labeled, disposable, plastic petri dish (7.48) or other similar container.

10.4.14 In order to ensure that an optimally-loaded filter is obtained, it is recommended that filters be prepared from several different aliquots of the dust suspension. For this series of filters, it is recommended that the volume of each aliquot of the original suspension be a factor of five higher than the previous one. If the filters are prepared in order of increasing aliquot volume, all of the filters for one sample can be prepared using one plastic disposable filtration unit, or without cleaning of glass filtration equipment between individual filtration. Before withdrawal of each aliquot from the sample, shake the suspension without additional sonification and allow to rest for 2 min.

10.4.15. There are many practical methods for drying MCE filters. The following are two examples that can be used: (1) dry MCE filters for at least 12 h (over desiccant) in an airtight cabinet-type desiccator (7.21); (2) to shorten the drying time (if desired), remove a plug of the damp filter and attach it to a glass slide (7.19) as described in 12.1.2 and 12.1.3. Place the slide with a filter plug or filter plugs (up to eight plugs can be attached to one slide) on a bed of desiccant, in the desiccator for 1 h.

10.4.16 PC filters do not require lengthy drying before preparation, but shall be placed in a desiccator for at least 30 min before preparation.

10.5 Prepare TEM specimens from small sections of each dried filter using the appropriate direct transfer preparation method.

11. Blanks

11.1 Prepare sample blanks that include both a process blank (50 mL of particle-free water) for each set of samples analyzed and one unused filter from each new box of sample filters (MCE or PC) used in the laboratory. If glass filtering units are used, prepare and analyze a process blank each time the filtering unit is cleaned. Blanks will be considered contaminated, if after analysis, they are shown to contain more than 53 asbestos structures per square millimetre. This generally corresponds to three or four asbestos structures found in ten grid openings. The source of the contamination must be found before any further analysis can be performed. Reject samples that were processed along with the contaminated blanks and prepare new samples after the source of the contamination is found.

11.2 Prepare field blanks which are included with sample sets in the same manner as the samples, to test for contamination during the sampling, shipping, handling, and preparation steps of the method.

12. TEM Specimen Preparation of Mixed Cellulose Ester (MCE) Filters

Norra 1—Use of either the accrone or the diamethylformamideacetic acid method is acceptable.

12.1 Acetone Fusing Method:

12.1.1 Remove a section (a plug) from any quadrant of the sample and blank filters. Sections can be removed from the filters using a 7 mm cork borer (7.32). The cork borer must be wet wiped after each time a section is removed.

12.1.2 Place the filter section (particle side up) on a clean microscope slide. Affix the filter section to the slide with a gummed page reinforcement (7.43), or other suitable means. Label the slide with a glass scribing tool or permanent marker (7.10).

12.1.3 Prepare a fusing dish from a glass petri dish (7.37) and a metal screen bridge (7.38) with a pad of five to six ashless paper filters (7.42) and place in the bottom of the petri dish (4). Place the screen bridge on top of the pad and saturate the filter pads with acetone. Place the slide on top of the bridge in the petri dish and cover the dish. Wait approximately 5 min for the sample filter to fuse and clear.

12.2 Dimethylformamide-Acetic Acid Method:

12.2.1 Place a drop of clearing solution that consists of 35 % dimethylformamide (DMF), 15 % glacial acetic acid, and 50 % Type II water (v/v) on a clean microscope slide. Gauge the amount used so that the cleaning solution just saturates the filter section.

12.2.2 Carefully lay the filter segment, sample surface upward, on top of the solution. Bring the filter and solution together at an angle of about 20° to help exclude air bubbles. Remove any excess clearing solution. Place the slide in an oven or on a hot plate, in a fume hood, at 65 to 70°C for 10 min.

12.3 Plasma etching of the collapsed filter is required.

12.3.1 The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher (7.27). Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the exact conditions that must be used. Insufficient etching will result in a failure to expose embedded fibers, and too much etching may result in the loss of particles from the filter surface. To determine the optimum time for ashing, place an unused 25 mm diameter MCE filter in the center of a glass microscope slide. Position the slide approximately in the center of the asher chamber. Close the chamber and evacuate to a pressure of approximately 40 Pa, while admitting oxygen to the chamber at a rate of 8 to 20 cm³/min. Adjust the tuning of the system so that the intensity of the plasma is maximized. Determine the time required for complete oxidation of the filter. Adjust the system parameters to achieve complete oxidation of the filter in a period of approximately 15 min. For etching of collapsed filters, use these operating parameters for a period of 8 min. For additional information on calibration, see the USEPA Asbestos-Containing Materials in Schools (4) or NIST/NYLAP Program Handbook for Airborne Ashestos Analysis (6) documents.

12.3.2 Place the glass slide containing the collapsed filters into the low-temperature plasma asher, and etch the filter.

12.4 Carbon coating of the collapsed and etched filters is required.

12.4.1 Carbon coating must be performed with a high-vacuum coating unit (7.4), capable of less than 10⁻⁴ torr (13 MPa) pressure. Units that are based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application and shall

not be used. Carbon rods (7.40) used for evaporators shall be sharpened with a carbon rod sharpener to a neck of about 4 mm in length and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 100 to 120 mm from the surface of the microscope slide held in the rotating device.

12.4.2 Place the glass slide holding the filters on the rotation device, and evacuate the evaporator chamber to a vacuum of at least 13 MPa. Perform the evaporation in very short bursts, separated by 3 to 4 s to allow the electrodes to cool. An alternate method of evaporation is by using a slow continuous applied current. An experienced analyst can judge the thickness of the carbon film to be applied. Conduct tests on unused filters first. If the carbon film is too thin, large particles will be lost from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen.

12.4.2.1 If the coating is too thick, it will lead to a TEM image that is lacking in contrast, and the ability to obtain electron diffraction patterns will be compromised. The carbon film shall be as thin as possible and still remain intact on most of the grid openings of the TEM specimen.

12.5 Preparation of the Jaffe Washer—The precise design of the Jaffe washer is not considered important, so any one of the published designs may be used (7, 8). One such washer consists of a simple stainless steel bridge contained in a glass petri dish.

12.5.1 Place several pieces of lens tissue (7.41) on the stainless steel bridge. The pieces of lens tissue shall be large enough to completely drape over the bridge and into the solvent. In a fume hood, fill the petri dish with acctone (or DMF) until the height of the solvent is brought up to contact the underside of the metal bridge as illustrated in Fig. 2.

12.6 Placing the Specimens into the Jaffe Washer:

12.6.1 Place the TEM grids (7.39) shiny side up on a piece of lens tissue or filter paper so that individual grids can be easily picked up with tweezers.

.12.6.2 Prepare three grids from each sample.

12.6.2.1 Using a curved scalpet blade (7.20), excise at least it two square (3 mm by 3 mm) pieces of the carbon-coated MCE filter from the glass slide.

12.6.2.2 Place the square filter piece carbon-side up on top of a TEM specimen grid.

12.6.2.3 Place the whole assembly (filter/grid) on the saturated lens tissue in the Jaffe washer.

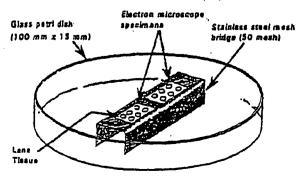


FIG. 2 Example of Design of Solvent Washer (Jatle Washer)

12.6.2.4 Place the three TEM grid sample filter preparations on the same piece of lens tissue in the Jaffe washer.

12.6.2.5 Place the lid on the Jaffe washer and allow the system to stand for several hours.

12.7 Alternately, place the grids on a low level (petri dish filled to the ½ mark) DMF Jaffe washer for 60 min. Add enough solution of equal parts DMF/acetone to fill the washer to the screen level. Remove the grids after 30 min if they have cleared, that is, all filter material has been removed from the carbon film, as determined by inspection in the TEM.

12.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean marked grid box.

13. TEM Specimen Preparation of Polycarbonate (PC)

13.1 Cover the surface of a clean microscope slide with two strips of double-sided adhesive tape.

13.2 Cut a strip of filter paper slightly narrower than the width of the slide. Position the filter paper strip on the center of the length of the slide.

13.3 Using a clean, curved scalpel blade, cut a strip of the PC filter approximately 25 by 6 mm. Use a rocking motion of the scalpel blade to avoid tearing the filter. Place the PC strip particle side up on the slide perpendicular to the long axis of the slide. The ends of the PC strip must contact the double sided adhesive tape. Each slide can hold several PC strips. With a glass marker, label each PC strip with the individual sample number.

13.4 Carbon coat the PC filter strips as discussed in 12.4.2. PC filters do not require exching.

Norg 2: Castion-Do not overheat the filter sections while carbon coating.

13.5 Prepare a Juste washer as described in 12.5, but fill the washer with chloroform or 1-methyl-2-pyrrolidone to the level of the screen.

13.6 Using a clean curved scalpel blade, excise three, 3-mm square filter pieces from each PC strip. Place the filter squares carbon side up on the shiny side of a TEM grid. Pick up the grid and filter section together and place them on the lens tissue in the Jaffe washer.

13.7 Place the lid on the Jaffe washer and rest the grids in place for at least 4 h. Best results are obtained with longer wicking times, up to 12 h.

13.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean, marked grid box.

14. Grid Opening Measurements

14.1 TEM grids must have a known grid opening area. Determine this area as follows:

14.2 Measure at least 20 grid openings in each of 20 random 75 to 100 µm (200-mesh) copper grids for a total of 400 grid openings for every 1000 grids used, by placing the 20 grids on a glass slide and examining them under the optical microscope. Use a calibrated graticule to measure the average length and width of the 20 openings from each of the individual grids. From the accumulated data, calculate the average grid opening area of the 400 openings.

14.3 Grid area measurements can also be made at the

TEM at a calibrated screen magnification of between 15 000 and 20 000X. Typically measure one grid opening for each grid examined. Measure grid openings in both the x and y directions and calculate the area.

14.4 Pre-calibrated TEM grids are also acceptable for this test method.

15. TEM Method

15.1 Microscope settings: 80 to 120 kV, 15 000 to 20 000X screen magnification for analysis (7.2).

15.2 Analyze two grids for each sample. Analyze one-half of the sample area on one sample grid preparation and the remaining half on a second sample grid preparation.

15.3 Determination of Specimen Suitability:

15.3.1 Carefully load the TEM grid, carbon side facing up (in the TEM column) with the grid bars oriented parallel/perpendicular to the length of the specimen holder. Use a hand lens or loupe, if necessary. This procedure will line up the grid with the X and y translation directions of the microscope. Insert the specimen holder into the microscope.

15.3.2 Scan the entire grid at low magnification (250X to 1000X) to determine its suitability for high magnification analysis as specified in 15.3.3.

15.3.3 Grids are acceptable for analysis if the following conditions are met:

15.3.3.1 The fraction of grid openings covered by the replica section is at least 50 %.

15.3.3.2 Relative to that section of the grid covered by the carbon replica, the fraction of intact grid openings is greater than 50 %.

15.3.3.3 The fractional area of undissolved filter is less than 10%.

15.3.3.4 The fraction of gaid openings with overlapping or folded replica film is less than 50 %.

15.3.3.5 At least 20 grid openings, that have no overlapping or folded replica, are less than 5 % covered with holes and have less than 5 % opaque area due to incomplete filter dissolution.

15.4 Determination of Grid Opening Suitability:

15.4.1 If the grid meets acceptance criteria, choose a grid opening for analysis from various areas of the grid so that the entire grid is represented. Determine the suitability of each individual grid opening prior to the analysis.

15.4.2 The individual grid opening must have less than 5 % holes over its area.

15.4.3 Grid openings must be less than 25 % covered with particulate matter.

15.4.4 Grid openings must be uniformly loaded.

15.5 Observe and record the orientation of the grid at 80 to 150X, on a grid map record sheet along with the location of the grid openings that are examined for the analysis. If indexed grids are used, a grid map is not required, but the identifying coordinates of the grid square must be recorded.

16. Recording Data Rules

16.1 Record on the count sheet any continuous grouping of particles in which an asbestos fiber is detected. Classify asbestos structures as fibers, bundles, clusters, or matrices as defined in 5.2.

16.2 Use the criteria for fiber, bundle, cluster, and matrix identification, as described in the USEPA Asbestos-Containing

Materials in Schools document (4). Record, for each AHERA structure identified, the length and width measurements.

16.3 Record NSD (No Structures Detected) when no structures are detected in the grid opening.

16.4 Identify structures classified as chrysotile identified by either electron diffraction or X-ray analysis (7.3) and recorded on a count sheet. Verify at least one out of every ten chrysotile structures by X-ray analysis.

16.5 Structures classified as amphiboles by X-ray analysis and electron diffraction are recorded on the count sheet. For more information on identification, see Yamate, et al. (7) or

Chatfield and Dillon (8).

16.6 Record a typical electron diffraction pattern for each type of asbestos observed for each group of samples (or a minimum of every five samples) analyzed. Record the micrograph number on the count sheet. Record at least one X-ray spectrum for each type of asbestos observed per sample. Attach the print-outs to the back of the count sheet. If the X-ray spectrum is stored, record the file and disk number on the count sheet.

16.7 Counting Rules:

16.7.1 At a screen magnification of between 15 000 and 20 000X evaluate the grids for the most concentrated sample loading; reject the sample if it is estimated to contain more than 50 asbestos structures per grid opening. Proceed to the next lower concentrated sample until a set of grids are obtained that have less than 30 asbestos structures per grid opening.

16.8 Analytical Sensitivity—An analytical sensitivity of approximately 1000 asbestos structures per square centimetre (calculated for the detection of a single asbestos structure) has been designed for this analysis. This sensitivity can be achieved by increasing the amount of liquid filtered, increasing the number of grid openings analyzed, or decreasing the size of the final filter. Occasionally, due to high particle loadings or high asbestos concentration, this analytical sensitivity cannot be practically achieved and stopping rules apply.

16.9 Limit of Detection—The limit of detection for this method is defined as, at a minimum, the counting of four asbestos structures during the TEM analysis. If less than four asbestos structures are counted during the analysis then the analysical result which will be reported will be less than the limit of detection and a "less than" sign (<) will appear before the number. All data shall be provided in the labo-

ratory report.

16.10 Stopping Rules:

16.10.1 The analysis is stopped upon the completion of the grid square that achieves an analytical sensitivity of less than 1000 ashestos structures per square centimetre.

16.10.2 If an analytical sensitivity of 1000 asbestos structures per square centimetre cannot be achieved after analyzing ten grid openings then stop on grid opening No. 10 or the grid opening which contains the 100th asbestos structure, whichever comes first. A minimum of four grid squares shall be analyzed for each sample.

16.10.2.1 If the analysis is stopped because of the 100th structure rule, the entire grid square containing the 100th structure must be counted.

16.11 After analysis, remove the grids from the TEM, and replace them in the appropriate grid storage holder.

17. Sample Storage

17.1 The washed-out sample cassettes can be discarded after use.

17.2 Sample grids and unused filter sections (7.18) must be stored for a minimum of one year.

18. Reporting

18.1 Report the following information for each dust sample analyzed:

18.1.1 Concentration in structures/cm².

18.1.2 The analytical sensitivity.

18.1.3 Types of asbestos present.

18.1.4 Number of asbestos structures counted.

18.1.5 Effective filtration area.

18.1.6 Average size of the TEM grid openings that were counted.

18.1.7 Number of grid openings examined.

18.1.8 Sample dilution used.

18.1:9 Area of the surface sampled.

18.1.10-Listing of size data for each structure counted.

18.1.11 A copy of the TEM count sheet or a complete listing of the caw data. An example of a typical count sheet is shown in Appendix X1.

13.2 Determine the amount of asbestos in any accepted sample using the following formula:

$$\frac{EFA \times 100 \text{ mL} \times \#STR}{GO \times GOA \times V \times SPL} = \text{asbestos structures/cm}^2 \qquad (1)$$

where:

#STR = number of asbestos structures counted,

EF.4 = effective filter area of the final sampling filter, mm²,

GO = number of grid openings counted,

GOA = average grid opening area, mm².

SPL = surface area sampled, cm², and

 volume of sample filtered in step 10.4.9, representing the actual volume taken from the original 100 mL suspension, mL.

19. Ouslity Control/Quality Assurance

19.1 In general, the laboratory's quality control checks are used to verify that a system is performing according to specifications regarding accuracy and consistency. In an analytical laboratory, spiked or known quantitative samples are normally used. However, due to the difficulties in preparing known quantitative asbestos samples, routine quality control testing focuses on re-analysis of samples (duplicate recounts).

19.1.1 Re-analyze samples at a rate of 1/10 of the sample sets (one out of every ten samples analyzed not including laboratory blanks). The re-analysis shall consist of a second

sample preparation obtained from the final filter.

19.2 In addition, quality assurance programs must follow the criteria shown in the USEPA Aspestos-Containing Materials in Schools document (4) and in the NIST/NVLAP Program Handbook for Airborne Aspestos Analysis document (6). These documents describe sample custody, sample preparation, blank checks for contamination, calibration, sample analysis, analyst qualifications, and technical facilities.

20. Calibrations

20.1 Perform calibrations of the instrumentation on a

regular basis, and retain these records in the laboratory, in accordance with the laboratory's quality assurance program.

20.2 Record calibrations in a log book along with dates of calibration and the attached backup documentation.

20.3 A calibration list for the instrument is as follows: 20.3.1 TEM:

20.3.1.1 Check the alignment and the systems operation. Refer to the TEM manufacturer's operational manual for detailed instructions.

20.3.1.2 Calibrate the camera length of the TEM in electron diffraction (ED) operating mode before ED patterns of unknown samples are observed. Camera length can be measured by using a carbon coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thick gold films will tend to mask weak diffraction spots from the fibrous particles. Since the unknown d-spacings of most interest in aspestos analysis are those which lie closest to the transmitted beam, multiple gold rings from thick films are unnecessary. Alternatively, a gold standard specimen can be used to obtain an average camera constant calculated for that particular instrument and can then be used for ED patterns of unknowns taken during the corresponding period.

20.3.1.3 Perform magnification calibration at the fluorescent screen. This calibration must be performed at the magnification used for structure counting. Calibration is performed with a grating replica (7.47) (for example, one containing at least 2160 lines/mm).

(a) Define a field of view on the fluorescent screen. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

(b) Frequency of calibration will depend on the service history of the particular microscope.

(c) Check the calibration after any maintenance of the microscope that involves adjustment of the power supply to the lens or the high voltage system or the mechanical disassembly of the electron optical column (apart from filament exchange).

(d) The analyst must ensure that the grating replica is placed at the same distance from the objective lens as the specimen.

(e) For instruments that incorporate a eucentric tilting specimen stage, all specimens and the grating replica must be placed at the eucentric position.

20.3.1.4 The smallest spot size of the TEM must be checked.

(a) At the crossover point, photograph the spot size at a screen magnification of 15 000 to 20 000X. An exposure time of 1 s is usually adequate.

(b) The measured spot size must be less than or equal to 250 nm.

20.4 EDXA:

20.4.1 The resolution and calibration of the EDXA must be verified.

20.4.1.1 Collect a standard EDXA Cu peak from the Cu grid.

20.4.1.2 Compare the X-ray energy versus channel

number for the Cu peak and be certain that readings are within ± 10 eV.

20.4.2 Collect a standard EDXA of crocidolite aspestos (NIST SRM 1866).

20.4.2.1 The elemental analysis of the crocidolite must resolve the Na peak.

20.4.3 Collect a standard EDXA of chrysotile ashestos.

20.4.3.1 The elemental analysis of chrysotile must resolve both Si and Mg on a single chrysotile fiber.

20.5 Ultrasonic bath calibration shall be performed as follows:

20.5.1 Fill the bath water to a level equal to the height of suspension in the glass sample container that will be used for the dust analysis. Operate the bath until the water reaches the equilibrium temperature.

20.5.2 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.3 Place the sample container in the water in the ultrasonic bath (with the power turned off). After 60 s, remove the glass container and record its temperature.

20.5.4 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.5 Place the second sample container into the water in the ultrasonic bath (with the power turned on). After 60 s, remove the glass container and record its temperature,

20.5.6 Calculate the rate of energy deposition into the sample container using the following formula:

$$R = 4.185 \times \sigma \times \rho \times \frac{(\theta_2 - \theta_1)}{l} \tag{2}$$

where

4.185 = Joules/cal,

R = energy deposition, watts/mL

= temperature rise with the ultrasonic bath not operating. *C.

 θ_1 = temperature rise with the ultrasonic bath operating, °C,

= time in seconds, 60 s (20.5.3 and 20.5.5),

 specific heat of the liquid in the glass sample container, 1.0 cal/g, and

ρ = density of the liquid in the glass sample container, 1.0 g/cm³.

20.5.7 Adjust the operating conditions of the bath so that the rate of energy deposition is in the range of 0.08 to 0.12 MW/m², as defined by this procedure.

21. Precision and Blas

21.1 Precision—The precision of the procedure in this test method is being determined using round robin data from participating laboratories.

21.2 Bias—Since there is no accepted reference material suitable for determining the bias of the procedure in this test method, bias has not been determined (see Specification D 3670).

Note 3—Round robin data is under development and will be presented as a research report.

22. Keywords

22.1 asbestos; microvacuuming; settled dust; TEM

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APPENDIX

(Nonmandatory Information)

XI. DUST SAMPLE ANALYSIS

XI.1 See Figs. XI.1 and XI.2 for the dust analysis worksheet and the TEM count sheet.

· DUST SAMPLE ANALYSIS

Clent		Accelerating Voltage	x			_
Sample ID:		indicated Mag:		. K	α	
Job Number:		Screen Mag:		K	X	
Date Sample Analyzed:	•	Microscope:	1	2	3	4 5
Number of Openings/Grids Counted:		Filter Type:		,		
Grid Accepted, 600X:	Yes No	Filter Size:		<u> </u>		
Percent Loading:	*	Filter Pore Size (µm):				
Grid Box #1:		Grid Opening:	1)	μπ	×_	μm
			2)	μπι	x _	· µm
Analyst:					•	
Reviewer:		Counting Rules:	AHERA	គោ	NET II	
Calculation Data:		•				
Effective Filter Area in mm2.		(EFA)		. %	···	
Number of Grid Openings Counter	d:	(GO)				
Avderage Grid Opening Area in m	m².	(GOA)				:
Volume of sample Filtered in ml:		(V)		•		
Surface area Sampled in cm ² :	•	(SPL)			•	
Number of Asbestos Structures Co	unted:" ((#STR)				
* If the number of asbestos structures could be provided by the number of asbestos structures could be provided by the number of asbestos structures: Continue of asbestos structures of the number of asbestos structures: Continue of asbestos structures of the number of asbestos structures:		S STRUCTURES "DUST res per cm²)			if detec	zion here.
	(Structures	per cm²)				

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Job Number:	
JOH MULLIPEL.	

Structure	Grid #			Length	Width		confirmati	on
#	Square #	Type	Structure	Microns		Morph	. SAED	EDS
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Note: Keys to Abbreviations Used in Figure:

		Type:		Str	ucture:		•	Others:
C	#	Chrysotlle	F	#	Fiber	NSD	=	No Structures Detected
AM	=	Amosite	В	-	Bundle	Marph	=	Morphalogy
CR	=	Crocidolite	C	=	Cluster	SAED	-	Selected Area Electron Diffraction
AC	26	Actinolite	М	=	Matrix	eds	.	Energy Dispersive X-Ray Spectroscopy
TR	=	Tremolite				ER	=	Inter-Row Spacing
AN	200	Anthophyllite				NP	=	No Pattern
N	==	Non Asbestos			•			

REFERENCES

- (1) Steel, E. and Wylie, A., "Mineralogical Characteristics of Asbestos," in Geology of Ashenos Deposits, Riordon, P. H., Ed., SME-AIME, 1981, pp. 93-101.
- (2) Zussman, J., "The Mineralogy of Asbestos," in Asbestos: Propertles, Applications and Hazards, John Wiley and Sons, 1979, pp. 45-67.
- (3) Leake, B. E., "Nomenclature of Amphiboles," American Mineralogist, Vol 63, 1978, pp. 1023-1052.
- (4) "USEPA Asbestos-Cantaining Materials in Schools Final Rule and Notice," Federal Register, 40 CFR Part 763, Appendix & to Sub-part E., Oct. 30, 1987.
- (5) OSHA, OSHA Technical Manual, OSHA Instruction CPL 2-208, Directorate of Technical Support, U.S. Department of Labor, Washington, DC 20210, Feb. 5, 1990, pp. 1-8 to 1-11.
- (6) NIST/NVLAP Program Handbook for Airborne Asbestos Analysis, NISTIR, August 1989, pp. 89–4137.
- (7) Yamate, G., Agarwall, S. C., and Gibbons, R. D., "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy," EPA Draft Report, Contract No. 68-02-1266, 1984.
- (8) Chatfield, E. J., and Dillon, M. J., "Analytical Method for the Determination of Asbestos in Water," EPA No. 600/4-83-043, 1983.

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Appendix C
Field Data Sheets

Sheet No.:	D
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LIBBY MONTANA FIELD SAMPLE DATA SHEET

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Scenario No	o.:	Field Lo	gbook No:		_Page No: _	San	npling Date:
Address:			· · · · · · · · · · · · · · · · · · ·	Ov	vner/Tenant:		
Land Use:	Residential	School	Commercial	Mining	Roadway	Other ()
Sampling T	Ceam: PES	CDM	Other	Name	s:		

Data Item	Cassette 1	Cassette 2	Cassette 3
Index ID			
Location ID			
Sample Group			
Location Description			
Category (circle)	FS Blank	FS Blank	FS Blank
Matrix Type (circle)	Building, Vehicle, NA Other	Building, Vehicle, NA Other	Building, Vehicle, NA Other
Vermiculite Present? (circle)	Yes No	Yes No	Yes No
Will samples be archived due to presence of vermiculite? (circle)	Yes No	Yes No	Yes No
Sample Area (cm²) (circle)	100 200 300	100 200 300	100 200 300
Filter Diameter (circle)	25mm 37mm	25mm 37mm	25mm 37mm
Pore Size (circle)	TEM45 PCM- 0.8	TEM 45 PCM- 0.8	TEM45 PCM- 0.8
Flow Meter Type (circle)	Rotometer Dry-Cal	Rotometer Dry-Cal	Rotometer Dry-Cal
Pump ID Number			
Flow Meter ID No.			
Start Date	·		
Start Time			
Start Flow (L/min)			
Stop Date			
Stop Time			
Stop Flow (L/min)			
Pump fault? (circle)	No Yes	No Yes	No Yes
Field Comments	100 cm ²	100 cm ²	100 cm ²
	100 cm ²	100 cm ²	100 cm ²
	100 cm ²	100 cm ²	100 cm ²
	Entered Validated	Entered Validated	Entered Validated

sheet	No ·	٧-	
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LIBBY MONTANA FIELD SAMPLE DATA SHEET PERSONAL AIR

	PERSONAL AIR				
Scenario No.:	Field Logbook No:	Page No:	Sampling Date:		
Address:		Owner/Tenant:			
Sampling Team: F	PES CDM Other	Names:			
Land Use: Resident	tial School Commercial	Mining Roadway Other	()		
Person Sampled:		SSN: Task:			
			~		
Data Item	Cassette 1	Cassette 2	Cassette 3		
Index ID					
Location ID					
Sample Group					
Location Description					
	·				
Category (circle)	FS Blank Rep	FS Blank Rep	FS Blank Rep		
Matrix Type (circle)	Indoor Outdoor NA	Indoor Outdoor NA	Indoor Outdoor NA		
Filter Diameter (circle)	25mm 37mm	25mm 37mm	25mm 37mm		
Pore Size (circle)	TEM45 PCM- 0.8	TEM45 PCM- 0.8	TEM45 PCM- 0.8		
Flow Meter Type (circle)	Rotometer DryCal	Rotometer DryCal	Rotometer DryCal		
Pump ID Number			<u></u>		
Flow Meter ID No.					
Start Date					
Start Time		·			
Start Flow (L/min)					
Stop Date			· · · · · · · · · · · · · · · · · · ·		
Stop Time					
Stop Flow (L/min)					
Pump fault?	No Yes	No Yes	No Yes		
MET Station onsite?	No Yes	No Yes	No Yes		
Sample Type	TWA EXC NA	TWA EXC NA	TWA EXC NA		
Field Comments					

Entered Validated

Validated

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Sheet No.: A-____

LIBBY MONTANA FIELD SAMPLE DATA SHEET STATIONARY AIR

Scenario No.:	Field Logbook No:	Page No:	Sampling Date:
Address:	·	Owner/Tenant	:
Land Use: Residential	School Commercial	Mining Roadway	Other ()
Sampling Team: PES	CDM Other	Names:	

Data Item	Cassette 1	Cassette 2	Cassette 3
Index ID			
Location ID			
Sample Group			
Location Description			
Category (circle)	FS Blank Rep	FS Blank Rep	FS Blank Rep
Matrix Type (circle)	Indoor Outdoor NA	Indoor Outdoor NA	Indoor Outdoor NA
Filter Diameter (circle)	25mm 37mm	25mm 37mm	25mm 37mm
Pore Size (circle)	TEM45 PCM- 0.8	TEM45 PCM- 0.8	TEM45 PCM- 0.8
Flow Meter Type (circle)	Rotometer DryCal	Rotometer DryCal	Rotometer DryCal
Pump ID Number			
Flow Meter ID No.			
Start Date			
Start Time			
Start Flow (L/min)			
Stop Date	11_		
Stop Time			
Stop Flow (L/min)			
Pump fault? (circle)	No Yes	No Yes	No Yes
MET Station onsite?	No Yes	No Yes	No Yes
Pre/Post (circle)	Pre Post Clear NA	Pre Post Clear NA	Pre Post Clear NA
Field Comments			
	Entered Validated	Entered Validated	Entered Validated

Appendix D

CDM SOP 4-1
Field Logbook Content and Control

SOP 4-1

Revision: 4

Date: June 20, 2001

Page 1 of 5

Prepared: Del Baird	Technical (Review: / Larry Davidson
QA Review: _ David O. Johnson	Approved. The Approved to be a below
Issued: lose hay J. Gustin 6/20/01	Signature Date
Issued: /Signature/Date	

1.0 OBJECTIVE

The objective of this standard operating procedure (SOP) is to set CDM Federal criteria for content entry and form of field logbooks. Field logbooks are an essential tool to document field activities for historical and legal purposes.

2.0 BACKGROUND

2.1 Definitions

Biota - The flora and fauna of a region.

<u>Magnetic Declination Corrections</u> - Compass adjustments to correct for the angle between magnetic north and geographical meridians.

2.2 Discussion

Information recorded in field logbooks includes field team names, observations, data, calculations, date/time, weather, and description of the data collection activity, methods, instruments, and results. Additionally, the logbook may contain deviations from plans and descriptions of wastes, biota, geologic material, and site features including sketches, maps, or drawings as appropriate.

3.0 RESPONSIBILITIES

Field Team Leader (FTL) - The FTL is responsible for ensuring that the format and content of data entries are in accordance with this procedure.

Site Personnel - All CDM Federal employees who make entries in field logbooks during onsite activities are required to read this procedure prior to engaging in this activity. The FTL will assign field logbooks to site personnel who will be responsible for their care and maintenance. Site personnel will return field logbooks to the records file at the end of the assignment.

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Revision: 4

Date: June 20, 2001

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4.0 REQUIRED EQUIPMENT

- Site-specific plans
- Field notebook
- Indelible black or blue ink pen
- Ruler or similar scale

5.0 PROCEDURES

5.1 Preparation

In addition to this SOP, site personnel responsible for maintaining logbooks must be familiar with all procedures applicable to the field activity being performed. These procedures should be consulted as necessary to obtain specific information about equipment and supplies, health and safety, sample collection, packaging, decontamination, and documentation. These procedures should be located at the field office.

Field logbooks shall be bound with lined, consecutively numbered pages. All pages must be numbered prior to initial use of the logbook. Prior to use in the field, each logbook will be marked with a specific document control number issued by the document control administrator, if required by the contract quality implementation plan (QIP). Not all contracts require document control numbers. The following information shall be recorded on the cover of the logbook:

- Field logbook document control number.
- Activity (if the logbook is to be activity-specific) and location.
- Name of CDM Federal contact and phone number(s).
- Start date
- In specific cases, special logbooks may be required (e.g., waterproof paper for storm water monitoring).

The first few (approximately five) pages of the logbook will be reserved for a table of contents (TOC). Mark the first page with the heading and enter the following:

TABLE OF CONTENTS

Date/Description

(Start Date)/Reserved for TOC 1-5

The remaining pages of the table of contents will be designated as such with "TOC" written on the top center of each page.

Page

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Revision: 4

Date: June 20, 2001

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5.2 Operation

The following is a list of requirements that must be followed when using a logbook:

- Record work, observations, quantities of materials, calculations, drawings, and related information directly in the logbook. If data collection forms are specified by an activity-specific plan, this information need not be duplicated in the logbook. However, any forms used to record site information must be referenced in the logbook.
- Do not start a new page until the previous one is full or has been marked with a single diagonal line so that additional entries cannot be made. Use both sides of each page.
- Do not erase or blot out any entry at any time. Indicate any deletion by a single line through the material to be deleted. Initial and date each deletion. Take care to not obliterate what was written previously.
- Do not remove any pages from the book.

Specific requirements for field logbook entries include:

- Initial and date each page.
- Sign and date the final page of entries for each day.
- Initial and date all changes.
- Multiple authors must sign out the logbook by inserting the following:

Above notes authored by:

- (Sign name)
- (Print name)
- (Date)
- A new author must sign and print his/her name before additional entries are made.
- Draw a diagonal line through the remainder of the final page at the end of the day.
- Record the following information on a daily basis:
 - Date and time
 - Name of individual making entry
 - Names of field team and other persons on site
 - Description of activity being conducted including station or location (i.e., well, boring, sampling location number) if appropriate
 - Weather conditions (i.e., temperature, cloud cover, precipitation, wind direction, and speed) and other pertinent data
 - Level of personal protection to be used
 - Serial numbers of instruments
 - Required calibration information
 - Serial/tracking numbers on documentation (e.g., carrier air bills)

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Revision: 4

Date: June 20, 2001

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Entries into the field logbook shall be preceded with the time (written in military units) of the observation. The time should be recorded frequently and at the point of events or measurements that are critical to the activity being logged. All measurements made and samples collected must be recorded unless they are documented by automatic methods (e.g., data logger) or on a separate form required by an operating procedure. In these cases, the logbook must reference the automatic data record or form.

At each station where a sample is collected or an observation or measurement made, a detailed description of the location of the station is required. Use a compass (include a reference to magnetic declination corrections), scale, or nearby survey markers, as appropriate. A sketch of station location may be warranted. All maps or sketches made in the logbook should have descriptions of the features shown and a direction indicator. It is preferred that maps and sketches be oriented so that north is toward the top of the page. Maps, sketches, figures, or data that will not fit on a logbook page should be referenced and attached to the logbook to prevent separation.

Other events and observations that should be recorded include:

- Changes in weather that impact field activities.
- Deviations from procedures outlined in any governing documents. Also record the reason for any noted deviation.
- Problems, downtime, or delays.
- Upgrade or downgrade of personal protection equipment.

5.3 Post-Operation

To guard against loss of data due to damage or disappearance of logbooks, completed pages shall be periodically photocopied (weekly, at a minimum) and forwarded to the field or project office. Other field records shall be photocopied and submitted regularly and as promptly as possible to the office. When possible, electronic media such as disks and tapes should be copied and forwarded to the project office.

At the conclusion of each activity or phase of site work, the individual responsible for the logbook will ensure that all entries have been appropriately signed and dated, and that corrections were made properly (single lines drawn through incorrect information, then initialed and dated). The completed logbook shall be submitted to the records file.

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Revision: 4

Date: June 20, 2001

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6.0 RESTRICTIONS/LIMITATIONS

Field logbooks constitute the official record of onsite technical work, investigations, and data collection activities. Their use, control, and ownership are restricted to activities pertaining to specific field operations carried out by CDM Federal personnel and their subcontractors. They are documents that may be used in court to indicate dates, personnel, procedures, and techniques employed during site activities. Entries made in these notebooks should be factual, clear, precise, and non-subjective. Field logbooks, and entries within, are not to be utilized for personal use.

7.0 REFERENCES

Sandia National Laboratories, Procedure for Preparing, Sampling and Analysis Plan, Site-Specific Sampling Plan, and Field Operating Procedures, QA-02-03, Albuquerque Environmental Program Department 3220, Albuquerque, New Mexico, 1991.

Sandia National Laboratories, Division 7723, Field Operation Procedure for Field Logbook Content and Control, Environmental Restoration Department, Albuquerque, New Mexico, 1992.

Appendix E

CDM SOP 2-1
Packaging and Shipping of Environmental Samples

SOP: 2-1 Revision: 1

Date: June 20, 2001

Page 1 of 27

Prepared: Krista Lippoldt	Technical Review: Brian Lenks 1
QA Review: <u>David O. Johnson</u>	Approved: Approved: Approved:
Issued: lose Hory J. Kustin 6/50/	Signature/Date

1.0 PACKAGING AND SHIPPING OF ALL SAMPLES – This standard operating procedure (SOP) applies to the packaging and shipping of all environmental samples. If the sample is preserved or radioactive, the following sections may also be applicable.

Section 2.0 - Packaging and Shipping of Samples Preserved with Hexane

Section 3.0 - Packaging and Shipping of Samples Preserved with Sodium Hydroxide

Section 4.0 - Packaging and Shipping of Samples Preserved with Hydrochloric Acid

Section 5.0 - Packaging and Shipping of Samples Preserved with Nitric Acid

Section 6.0 - Packaging and Shipping of Samples Preserved with Sulfuric Acid

Section 7.0 - Packaging and Shipping of Limited Quantity Radioactive Samples

1.1 OBJECTIVE

The objective of this SOP is to outline the requirements for the packaging and shipment of environmental samples.

1.2 BACKGROUND

1.2.1 Definitions

<u>Environmental Sample</u> - An environmental sample is any sample that has less than reportable quantities for any hazardous constituents according to Department of Transportation (DOT) regulations promulgated in 49 CFR - Part 172.

<u>Custody Seal</u> – A custody seal is a narrow adhesive-backed seal that is applied to individual sample containers and/or the sample shipping container (i.e. cooler) before offsite shipment. Custody seals are used as a protective mechanism to ensure that sample integrity is not compromised during transportation from the field to the analytical laboratory.

<u>Secondary Containment</u> – A secondary containment is the container that the sample is shipped in (i.e., plastic overpackaging if liquid sample is collected in glass).

Exempted Quantity – Exempted quantity is the amount of hazardous material that does not fall under DOT/IATA/ICAO regulations. This exemption is very difficult to meet; most shipments will be made under limited quantity.

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Date: June 20, 2001

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<u>Limited Quantity</u> – Limited quantity is the maximum amount of a hazardous material for which there is a specific labeling or packaging exception.

<u>Performance Testing</u> - Performance testing is the required testing of outer packaging. These tests include the drop and stacking test.

<u>Qualified Shipper</u> – A qualified shipper is a person who has been adequately trained to perform the functions of shipping hazardous materials.

1.2.2 Discussion

Proper packaging and shipping is necessary to ensure the protection of the integrity of environmental samples shipped for analysis.

1.2.3 Associated Procedure

• CDM Federal SOP 1-2, Sample Custody

1.3 RESPONSIBILITIES

Field Team Leader (FTL) - The field team leader is responsible for ensuring that packaging and sampling procedures are conducted in accordance with this SOP. The field team leader is also responsible for ensuring that CDM Federal properly coordinates laboratory analysis of samples.

1.4 REQUIRED EQUIPMENT

- Coolers with return address of CDM Federal office
- Heavy-duty plastic garbage bags
- Plastic Ziploc®-type bags, small and large
- Clear tape
- Fiber tape nylon reinforced strapping tape
- Duct tape
- Vermiculite (or equivalent)*
- Bubble wrap (optional)
- Ice
- Custody seals
- Completed chain-of-custody record or CLP custody records, if applicable
- Completed bill of lading
- "This End Up" and directional arrow labels
- * Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

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1.5 PROCEDURES

The following steps must be followed when packing sample bottles and jars for shipment:

- 1. Verify the samples undergoing shipment meet the definition of "Environmental Sample" and are not a hazardous material as defined by DOT. Professional judgment and/or consultation with the appropriate health and safety coordinator or the health and safety manager should be observed.
- 2. Select a sturdy cooler in good repair. Secure and tape the drain plug with fiber or duct tape. Line the cooler with a large heavy-duty plastic garbage bag.
- 3. Be sure the caps on all bottles are tight (will not leak); check to see that labels and chain-of-custody records are completed properly (SOP 1-2, Sample Custody).
- 4. Place all bottles in separate and appropriately sized plastic zip-top bags and close the bags. Up to three VOA vials may be packed in one bag. Bottles may be wrapped in bubble wrap. Optionally, place three to six VOA vials in a quart metal can and then fill the can with vermiculite or equivalent. Note: Trip blanks must be included in coolers containing VOA samples.
- 5. Place 2 to 4 inches of vermiculite (or equivalent) into a cooler that has been lined with a garbage bag, and then place the bottles and cans in the bag with sufficient space to allow for the addition of more packing material between the bottles and cans. It is preferable to place glass sample bottles and jars into the cooler vertically. Due to the strength properties of a glass container, there is much less chance for breakage when the container is packed vertically rather than horizontally.
- 6. Put ice in large plastic zip-top bags (double bagging the zip-tops is preferred) and properly seal. Place the ice bags on top of and/or between the samples. Several bags of ice are required (dependant on outdoor temperature, staging time, etc.) to maintain the cooler temperature at approximately 4° centigrade. Fill all remaining space between the bottles or cans with packing material. Securely fasten the top of the large garbage bag with fiber or duct tape.
- 7. Place the completed chain-of-custody record or the CLP traffic report form (if applicable) for the laboratory into a plastic zip-top bag, seal the bag, tape the bag to the inner side of the cooler lid and close the cooler.
- 8. The cooler lid shall be secured with nylon reinforced strapping tape by wrapping each end of the cooler a minimum of two times. Attach a completed chain-of-custody seal across the hinges of the cooler on opposite sides. The custody seals should be affixed to the cooler with half of the seal on the strapping tape so that the cooler cannot be opened without breaking the seal. Complete two more wraps around with fiber tape and place clear tape over the custody seals.

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9. The shipping container lid must be marked "THIS END UP" and arrow labels that indicate the proper upward position of the container should be affixed to the cooler. A label containing the name and address of the shipper (CDM Federal) shall be placed on the outside of the container. Labels used in the shipment of hazardous materials (such as Cargo Only Air Craft, Flammable Solids, etc.) are not permitted on the outside of containers used to transport environmental samples and shall not be used. The name and address of the laboratory shall be placed on the container, or when shipping by common courier, the bill of lading shall be completed and attached to the lid of the shipping container.

1.6 RESTRICTIONS/LIMITATIONS

The holding times for the samples packed for shipment must not be exceeded. It is recommended that samples be packed in time to be shipped nightly for overnight delivery. Use caution when shipping samples for weekend delivery; make arrangements with the laboratory before sending samples.

2.0 PACKAGING AND SHIPPING OF SAMPLES PRESERVED WITH HEXANE

2.1 OBJECTIVE

This section provides guidance for the shipment of soil and water environmental samples regulated under the DOT Hazardous Materials Regulations and the IATA/ICAO Dangerous Goods Regulations for shipment by air and applies only to domestic shipments.

2.2 BACKGROUND

2.2.1 Definitions

Section 1.2.1 defines the terms relevant to this section.

2.2.2 Transportation

This section was prepared for the shipment of hexane-preserved samples.

2.2.3 Containers

40 ml glass VOA vials (up to 1L per outer package)

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2.3 RESPONSIBILITY

It is the responsibility of the qualified shipper to ensure that each shipment contains no more than the maximum of 24 VOA vials for a total liquid volume of 1 liter and that the shipment is packaged according to IATA/ICAO packaging instruction Y305 for limited quantities of hexane.

REQUIRED EQUIPMENT

- Outer packaging (for limited quantities) insulated cooler that has passed the performance test
- Garbage bags
- Clear tape
- Duct tape
- Strapping tape (optional)
- Ziploc®-type bags, small and large
- Vermiculite (or equivalent)*
- Bubble wrap
- Ice
- Chain-of-custody seals
- Chain-of-custody form
- Survey documentation (if shipping from Department of Energy [DOE] or radiological sites)
- Class 3 flammable liquid labels
- Orientation labels
- Consignor/consignee labels
- * Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

2.5 PACKAGING

The following steps are to be followed when packaging limited quantity samples shipments.

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape prior to sampling.
- At a minimum the label must contain:
 - Project name
 - Project number
 - Date and time of sample collection
 - Sample location
 - Sample identification number
 - Collector's initials

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- Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody)
- Wrap each container (40 ml VOA vials) in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble wrapped container into a 2.7 mil Ziploc®-type bag, removing trapped air.
- Place wrapped containers inside a polyethylene bottle filled with vermiculite; seal the bottle. (Maximum of 4 VOA vials will fit inside a 500-ml wide-mouth polyethylene bottle.)
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tying or taping.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a Ziploc®-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

HEXANES MIXTURE UN1208 LTD. QTY.

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Flammable Liquid label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marking locations is shown in Figure 1.

NOTE: No marking or labeling can be obscured by strapping or duct tape.

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NOTE: The inner packaging of dangerous goods may be placed into the designated cooler for shipment. Other non-regulated environmental samples may be added to the cooler for shipment.

- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure the exterior surfaces do not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

Strapping
Tape

Hexanes Mixture
UN1208
LTD. QTY.

Taped
Drain

Orientation Labels

Figure 1 Example of Cooler Label/Marking Locations

3.0 PACKAGING AND SHIPPING OF SAMPLES PRESERVED WITH SODIUM HYDROXIDE

3.1 OBJECTIVE

This section provides guidance for the shipment of soil and water environmental samples regulated under the DOT Hazardous Materials Regulations and the IATA/ICAO Dangerous Goods Regulations for shipment by air and applies only to domestic shipments.

Hazard Class Label

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3.2 BACKGROUND

3.2.1 Definitions

Section 1.2.1 defines the terms relevant to this section.

3.2.2 Transportation

This section was prepared for the shipment of sodium hydroxide (NaOH) preserved samples.

3.2.3 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Exempted Quantities of Preservatives

Preserv	ative	Desired in Final Quantity of Preservative (ml) Sample Container					ıl) for Spec	ified
		pН	Conc.	40 ml	125 ml	250 ml	500 ml	1 L
NaOH	30%	>12	0.08%		.25	0.5	1	2

5 drops = 1 ml

3.3 RESPONSIBILITY

It is the responsibility of the qualified shipper to determine the amount of preservative in each sample so that accurate determination of quantities can be made.

REQUIRED EQUIPMENT

- Outer packaging (for limited quantities) insulated cooler that has passed the performance test.
- Garbage bags
- Clear tape
- Duct tape
- Strapping tape (optional)
- Ziploc®-type bags, small and large
- Vermiculite (or equivalent)*
- Bubble wrap (optional)
- Ice
- Custody seals
- Chain-of-custody form

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- Survey documentation (if shipping from Department of Energy [DOE] or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels
- * Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

3.5 PACKAGING

Samples containing NaOH as a preservative that exceed the exempted concentration of 0.08 percent (2 ml of a 30 percent per liter) will be shipped as a limited quantity per packing instruction Y809 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are to be followed when packaging limited quantity samples shipments.

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape prior to sampling.
- At a minimum the label must contain:
 - Project name
 - Project number
 - Date and time of sample collection
 - Sample location
 - Sample identification number
 - Collector's initials
 - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody)
- This step is optional; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble wrapped container into a 2.7 mil Ziploc®-type bag, removing trapped air.
- Place glass containers inside a polyethylene bottle filled with vermiculite; seal the bottle.
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tying or taping.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited quantity shipment of dangerous goods.

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- Secure the chain-of-custody form (placed inside a Ziploc®-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

SODIUM HYDROXIDE SOLUTION UN1824 LTD. QTY.

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Corrosive label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marking locations is shown in Figure 1.
 - NOTE: Samples meeting the exemption concentration of 0.08 percent NaOH by weight will be shipped as non-regulated or non-hazardous.
 - NOTE: No marking or labeling can be obscured by strapping or duct tape.
 - NOTE: The inner packaging of dangerous goods may be placed into the designated cooler for shipment. Other non-regulated environmental samples may be added to the cooler for shipment.
- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure the exterior surfaces do not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

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4.0 PACKAGING AND SHIPPING OF SAMPLES PRESERVED WITH HYDROCHLORIC ACID

4.1 OBJECTIVE

This section provides guidance for the shipment of soil and water environmental samples regulated under the DOT Hazardous Materials Regulations and the IATA/ICAO Dangerous Goods Regulations for shipment by air and applies only to domestic shipments.

4.2 BACKGROUND

4.2.1 Definitions

Section 1.2.1 defines the terms relevant to this section.

4.2.2 Transportation

This section was prepared for the shipment of hydrochloric acid (HCI) preserved samples.

4.2.3 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Exempted quantities of preservatives

Preservative		3	l in Final mple	Quantity of Preservative (ml) for Specified Container				
		pН	Conc.	40 ml	125 ml	250 ml	500 ml	1 L
HCl	2N	<2	0.04%	.2	.5	1		

5 drops = 1 ml

4.3 RESPONSIBILITY

It is the responsibility of the qualified shipper to determine the amount of preservative in each sample so that accurate determination of quantities can be made.

4.4 REQUIRED EQUIPMENT

- Outer packaging (for limited quantities) insulated cooler that has passed the performance test.
- Garbage bags
- Clear tape

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- Duct tape
- Strapping tape (optional)
- Ziploc®-type bags, small and large
- Vermiculite (or equivalent)*
- Bubble wrap
- Ice
- Custody seals
- Chain-of-custody form
- Survey documentation (if shipping from Department of Energy [DOE] or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels
- * Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

4.5 PACKAGING

The following steps are to be followed when packaging limited quantity samples shipments.

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape prior to sampling.
- At a minimum the label must contain:
 - Project name
 - Project number
 - Date and time of sample collection
 - Sample location
 - Sample identification number
 - Collector's initials
 - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody)
- Wrap each container (40 ml VOA vials) in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble wrapped container into a 2.7 mil Ziploc®-type bag, removing trapped air.
- Place wrapped containers inside a polyethylene bottle filled with vermiculite; seal the bottle. (Maximum of 4 VOA vials will fit inside a 500-ml wide-mouth polyethylene bottle.)
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.

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- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tying or taping.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a Ziploc®-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. OTY. (as shown below).

HYDROCHLORIC ACID SOLUTION UN1789 LTD. QTY.

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Corrosive label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marking locations is shown in Figure 1.
 - **NOTE**: Samples meeting the exemption concentration of 0.04 percent HCl by weight will be shipped as non-regulated or non-hazardous.
 - **NOTE**: No marking or labeling can be obscured by strapping or duct tape.
 - **NOTE**: The inner packaging of dangerous goods may be placed into the designated cooler for shipment. Other non-regulated environmental samples may be added to the cooler for shipment.
- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure the exterior surfaces do not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.

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- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

5.0 PACKAGING AND SHIPPING OF SAMPLES PRESERVED WITH NITRIC ACID

5.1 OBJECTIVE

This section provides guidance for the shipment of soil and water environmental samples regulated under the DOT Hazardous Materials Regulations and the IATA/ICAO Dangerous Goods Regulations for shipment by air and applies only to domestic shipments.

5.2 BACKGROUND

5.2.1 Definitions

Section 1.2.1 defines the terms relevant to this section.

5.2.2 Transportation

This section was prepared for the shipment of nitric acid (HNO₃) preserved samples.

5.2.3 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Exempted quantities of preservatives

Preserv	ative	_	l in Final nple	Quantity of Preservative (ml) for Specif Container				
		pН	Conc.	40 ml	125 ml	250 ml	500 ml	1 L
HNO₃	6N	<2	0.15%	,	2	4	5	8

5 drops = 1 ml

5.3 RESPONSIBILITY

It is the responsibility of the qualified shipper to determine the amount of preservative in each sample so that accurate determination of quantities can be made.

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5.4 REQUIRED EQUIPMENT

- Outer packaging (for limited quantities) insulated cooler that has passed the performance test.
- Garbage bags
- Clear tape
- Duct tape
- Strapping tape (optional)
- Ziploc®-type bags, small and large
- Vermiculite (or equivalent)*
- Bubble wrap (optional)
- Ice
- Custody seals
- Chain-of-custody form
- Survey documentation (if shipping from Department of Energy [DOE] or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels
- * Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

5.5 PACKAGING

Samples containing HNO₃ as a preservative that exceed the exempted concentration of 0.15% HNO₃ will be shipped as a limited quantity per packing instruction Y807 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are to be followed when packaging limited quantity samples shipments.

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape prior to sampling.
- At a minimum the label must contain:
 - Project name
 - Project number
 - Date and time of sample collection
 - Sample location
 - Sample identification number
 - Collector's initials
 - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody)

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- This step is optional; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble wrapped container into a 2.7 mil Ziploc®-type bag, removing trapped air.
- Place glass containers inside a polyethylene bottle filled with vermiculite; seal the bottle.
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tying or taping.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a Ziploc®-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

NITRIC ACID SOLUTION (with less then 20%) UN2031 LTD. OTY.

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Corrosive label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marking locations is shown in Figure 1.

NOTE: Samples meeting the exemption concentration of 0.15 percent HNO₃ by weight will be shipped as non-regulated or non-hazardous.

NOTE: No marking or labeling can be obscured by strapping or duct tape.

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NOTE:

The inner packaging of dangerous goods may be placed into the designated cooler for shipment. Other non-regulated environmental samples may be added to the cooler for shipment.

- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure the exterior surfaces do not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

6.0 PACKAGING AND SHIPPING OF SAMPLES PRESERVED WITH SULFURIC ACID

6.1 **OBJECTIVE**

This section provides guidance for the shipment of soil and water environmental samples regulated under the DOT Hazardous Materials Regulations and the IATA/ICAO Dangerous Goods Regulations for shipment by air and applies only to domestic shipments.

6.2 **BACKGROUND**

6.2.1 Definitions

Section 1.2.1 defines the terms relevant to this section.

6.2.2 Transportation

This section was prepared for the shipment of sulfuric acid (H₂SO₄) preserved samples.

6.2.3 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Exempted quantities of preservatives

Preservative		Desired in Final Sample		Quantity of Preservative (ml) for Specified Container				ified
		рН	Conc.	40 ml	125 ml	250 ml	500 ml	1 L
H ₂ SO ₄	37N	<2	0.35%	.1	.25	0.5	1	2
				drops = 1	mi			

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6.3 RESPONSIBILITY

It is the responsibility of the qualified shipper to determine the amount of preservative in each sample so that accurate determination of quantities can be made.

6.4 REQUIRED EQUIPMENT

- Outer packaging (for limited quantities) insulated cooler that has passed the performance test.
- Garbage bags
- Clear tape
- Duct tape
- Strapping tape (optional)
- Ziploc®-type bags, small and large
- Vermiculite (or equivalent)*
- Bubble wrap
- Ice
- Custody seals
- Chain-of-custody form
- Survey documentation (if shipping from Department of Energy [DOE] or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels
- * Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

6.5 PACKAGING

Samples containing H₂SO₄ as a preservative that exceed the exempted concentration of 0.35 percent will be shipped as a limited quantity per packing instruction Y809 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are to be followed when packaging limited quantity samples shipments.

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape prior to sampling.
- At a minimum the label must contain:
 - Project name
 - Project number
 - Date and time of sample collection

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- Sample location
- Sample identification number
- Collector's initials
- Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody)
- Wrap each glass container in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble wrapped container into a 2.7 mil Ziploc®-type bag, removing trapped air.
- Place glass containers inside a polyethylene bottle filled with vermiculite; seal the bottle.
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tying or taping.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a Ziploc®-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

SULFURIC ACID SOLUTION UN2796 LTD. QTY.

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Corrosive label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marking locations is shown in Figure 1.

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NOTE:

Samples meeting the exemption concentration of 0.35 percent H₂SO₄ by weight

will be shipped as non-regulated or non-hazardous.

NOTE:

No marking or labeling can be obscured by strapping or duct tape.

NOTE:

The inner packaging of dangerous goods may be placed into the designated cooler

for shipment. Other non-regulated environmental samples may be added to the

cooler for shipment.

• When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure the exterior surfaces do not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.

• Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited Quantity (Appendix A).

Complete a Dangerous Goods Airbill.

7.0 PACKAGING AND SHIPPING OF LIMITED QUANTITY RADIOACTIVE SAMPLES

7.1 OBJECTIVE

This section provides guidance for the shipment of soil and water environmental samples regulated under the DOT Hazardous Materials Regulations and the IATA/ICAO Dangerous Goods Regulations for shipment by air and applies only to domestic shipments.

7.2 BACKGROUND

7.2.1 Definitions

Section 1.2.1 defines the terms relevant to this section.

7.2.2 Transportation

This section was prepared for the shipment of environmental samples containing radioactive materials in limited quantities.

7.2.3 Containers

The inner packaging containers that may be used for these shipments include:

Any size sample container

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7.3 DESCRIPTION/RESPONSIBILITIES

- The qualified shipper will ship all samples that meet the Class 7 definition of radioactive materials and meet the activity requirements specified in Table 7 of 49 CFR 173.425, as Radioactive Materials in Limited Quantity. The qualified shipper will verify that all packages and their contents meet the requirements of 49 CFR 173.421, "Limited Quantities of Radioactive Materials."
- The packaging used for shipping will meet the general requirements for packaging and packages specified in 49 CFR 173.24 and the general design requirements provided in 173.410. These standards state that a package must be capable of withstanding the effects of any acceleration, vibration, or vibration resonance that may arise under normal condition of transport without any deterioration in the effectiveness of the closing devices on the various receptacles or in the integrity of the package as a whole and without loosening or unintentionally releasing the nuts, bolts, or other securing devices even after repeated use.
- If the shipment is from a Department of Energy (DOE) facility, radiological screenings will be completed on all samples taken. The qualified shipper will review the results of each screening (alpha, beta, and gamma speciation). Samples will not be shipped offsite until the radiological screening has been performed.
- The total activity for each package will not exceed the relevant limits listed in Table 7 of 49 CFR 173.425. The A₂ value of the material will be calculated based on all radionuclides found during previous investigations (if any) in the area from which the samples are derived. The A₂ values to be used will be the most restrictive of all potential radionuclides as listed in 49 CFR 173.435.
- The radiation level at any point on the external surface of the package bearing the sample(s) will not exceed 0.005 mSv/hour (0.5 mrem/hour). These will be verified by dose and activity monitoring prior to shipment of the package.
- The removable radioactive surface contamination on the external surface of the package will not exceed the limits specified in 49 CFR 173.443(a). CDM Federal will use the DOE-established free release criteria for removable surface contamination of less than 20 dpm/100 cm² (alpha) and 1000 dpm/100 cm² (beta/gamma). It should be noted that these values are more conservative than the DOT requirements for removable surface contamination.
- The qualified shipper will verify that the outside of the inner packaging is marked "Radioactive".
- The qualified shipper will verify that the excepted packages prepared for shipment under the provisions of 49 CFR 173.421 have a notice enclosed, or shown on the outside of the package, that reads, "This package conforms to the conditions and limitations specified in 49 CFR 173.421 for radioactive material, excepted package-limited quantity of material, UN2910".

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7.4 REQUIRED EQUIPMENT

- Cooler or other acceptable outer packaging
- Garbage bags
- Clear tape
- Duct tape
- Strapping tape (optional)
- Ziploc®-type bags, small and large
- Vermiculite (for water samples) or equivalent*
- Bubble wrap (optional)
- Ice (if necessary)
- Custody seals
- Chain-of-custody form
- Survey documentation/radiation screening results (if shipping from DOE or radiological sites)
- Orientation labels
- Exempted quantities label
- Consignor/consignee labels
- * Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

7.5 PACKAGING

The following steps are to be followed when packaging limited quantity samples shipments.

- The cooler is to be surveyed by a qualified radiation control technician to ensure the exterior surfaces do not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape prior to sampling.
- At a minimum the label must contain:
 - Project name
 - Project number
 - Date and time of sample collection
 - Sample location
 - Sample identification number
 - Collector's initials
- This step is optional; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.

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- Place sufficient amount of vermiculite, or approved packaging material, in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- If required, place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tying or taping.
- Place a label marked "Radioactive" on the outside of the sealed bag.
- Enclose a notice that includes the name of the consignor or consignee and the following statement: "This package conforms to the conditions and limitations specified in 49 CFR 173.421 for radioactive material, excepted package-limited quantity of material, UN2910.
- The maximum weight of the package shall not exceed 30 kg (66 lbs) for any limited quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a Ziploc®-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- If a cooler is used, wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix package orientation labels on two opposite sides of the cooler/package.
- Affix a completed Excepted Quantities label to the side of the cooler/package.
- Secure any marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of the cooler labeling/marking is shown in Figure 2.

NOTE: No marking or labeling can be obscured by strapping or duct tape.

• Complete the Shipment Quality Assurance Checklist (Appendix B).

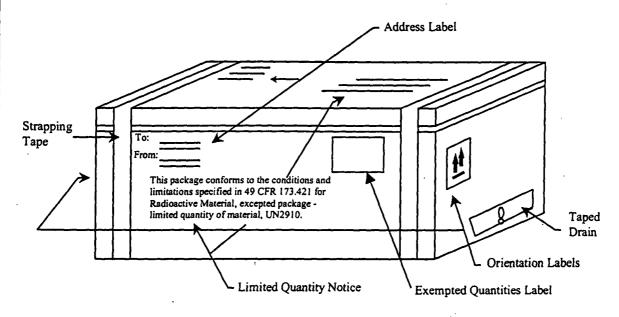
NOTE: Except as provided in 49 CFR 173.426, the package will not contain more than 15 grams of ²³⁵U.

NOTE: A declaration of dangerous goods is not required.

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Figure 2 Radioactive Material - Limited Quantity Cooler Marking Example



8.0 REFERENCES

U.S. Environmental Protection Agency, Sampler's Guide to the Contract Laboratory Program, EPA/540/P-90/006, December 1990.

U.S. Environmental Protection Agency, Region IV, Standard Operating Procedures and Quality Assurance Manual, February 1991.

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APPENDIX A Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited Quantity

Sam	ole Pac	kaging	
Yes	No	N/A	•
			The VOA vials are wrapped in bubble wrap and placed inside a Ziploc®-type bag.
			The VOA vials are placed into a polyethylene bottle, filled with vermiculite, and tightly sealed.
			The drain plug is taped inside and outside to ensure control of interior contents.
0 .			The samples have been placed inside garbage bags with sufficient bags of ice to preserve samples at 4°C.
0			The cooler exceeds the 66-pound limit for limited quantity shipment.
٥	۵		The garbage bag has been sealed with tape (or tied) to prevent movement during shipment.
0			The chain-of-custody has been secured to the interior of the cooler lid.
	ā		The cooler lid and sides have been taped to ensure a seal.
٥			The custody seals have been placed on both the front and back hinges of the cooler, using waterproof tape.
<u>Air V</u>	Vaybill	Completi	<u>ion</u>
Yes	No	N/A	
ם	٥		Section 1 has the shipper's name, company and address; the account number, date, internal billing reference number; and the telephone number where the shipper can be reached.
			Section 2 has the recipient's name and company along with a telephone number where they can be reached.
			Section 3 has the Bill Sender box checked.
a			Section 4 has the Standard Overnight box checked.
			Section 5 has the Deliver Weekday box checked.
۵			Section 6 has the number of packages and their weights filled out. Was the total of all packages and their weights figured up and added at the bottom of Section 6?
Q		0	Under the Transport Details box, the Cargo Aircraft Only box is obliterated, leaving only the Passenger and Cargo Aircraft box.
		a	Under the Shipment Type, the Radioactive box is obliterated, leaving only the Non-Radioactive box.
Q		a	Under the Nature and Quantity of Dangerous Goods box, the Proper Shipping Name,
			Class or Division, UN or ID No., Packing Group, Subsidiary Risk, Quantity and Type of Packing, Packing Instructions and Authorization have been filled out for the type of chemical being sent.
	۵		The Name, Place & Date, Signature, and Emergency Telephone number appears at the bottom of the FedEx Airbill.
	a	۵	The statement "In accordance with IATA/ICAO" appears in the Additional Handling Information box.
			J

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Proper Shipping Name	Class or Division	UN or ID No.	Packing Group	Sub Risk	Quantity	Packing Instruction	Authorization
Hydrochloric Acid Solution	8	UN1789	II		l plastic box × 0.5 L	Y809	LTD QTY
Nitric Acid Solution (with less than 20%)	8	UN2031	II		l plastic box × 0.5 L	Y807	LTDQTY
Sodium Hydroxide Solution	8	UN1824	II.		1 plastic box × 0.5 L	Y809	LTD QTY
Sulfuric Acid Solution	8	UN2796	II		l plastic box × 0.5 L	Y809	LTD QTY
Hexanes	3	UN1208	11		l plastic box × 1 L	Y305	LTD QTY

Sample Cooler Labeling

Yes	No	N/A	
ם		Ō	The proper shipping name, UN number, and LTD. QTY. appears on the shipping container.
۵		a	The corresponding hazard labels are affixed on the shipping container; the labels are not obscured by tape.
		0	The name and address of the shipper and receiver appear on the top and side of the shipping container.
			The air waybill is attached to the top of the shipping container.
			Up Arrows have been attached to opposite sides of the shipping container.
			Packaging tape does not obscure markings or labeling.

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APPENDIX B SHIPMENT QUALITY ASSURANCE CHECKLIST

Date:			Shipper: Destination:	
Item(s) D	Descrip	tion		
Radionuc	clide(s)):		
Radiolog	ical Su	ırve	Results: surfacemrem/hr 1 meter	
Instrumer	nt Use	d: N	Ifgr: Model:	
S/N:			Cal Date:	
			LIMITED QUANTITY OR INSTRUMENT AND ARTICLE	
Yes		 3. 4. 6. 7. 8. 	Strong tight package (package that will not leak material during conditions normall incidental to transportation). Radiation levels at any point on the external surface of package less than or equal to 0. mrem/hr. Removable surface contamination less than 20 dpm/100 cm² (alpha) and 1000 dpm/100 cm (beta/gamma). Outside inner package bears the marking "Radioactive". Package contains less than 15 grams of 235U (check yes if 235U not present). Notice enclosed in or on the package that includes the consignor or consignee and the statement, "This package conforms to the conditions and limitations specified in 49 CFR 173.421 for radioactive material, excepted package-limited quantity of material, UN2910." Activity less than that specified in 49 CFR 173.425. Permissible package limit: Package Quantity: On all air shipments, the statement, Radioactive Material, excepted package-limited quantity of material shall be noted on the air waybill.	5 1 ²
Qualified	Shipp	er:	Signature:	

Appendix F

CDM SOP 1-2 Sample Custody

Project-Specific Modification

Reason for and duration of modification: Sample custody procedures for the Libby asbestos project vary slightly from SOP 1-2. These modifications are necessary for the entire duration of the project.

Sample custody for all samples will be in accordance with SOP 1-2, with the following modifications:

<u>Section 3.0, Responsibilities</u> - The field sample custodian is referred to as the sample coordinator for the Libby Asbestos Project.

<u>Section 4.0, Required Supplies</u> - A project-specific chain-of-custody (COC) form will be used for the Libby Asbestos project.

<u>Section 5.1, Chain-of-Custody Record</u> – The project-specific COC form will be completed according to the following guidelines:

Send to: Name of the laboratory that will receive the samples specific to COC. To be completed by the sample coordinator.

Via: Hand delivery or shipped. Hand delivery refers to samples delivered by hand to the onsite laboratory; shipped refers to samples sent to the laboratory by delivery service (i.e., Federal Express). To be completed by the sample coordinator.

Project: All samples collected in accordance with this sampling and analysis plan (SAP) are part of the CSS. Circle CSS. To be completed by the field team.

Sample Placed in Cooler/Bag: Refers to visual confirmation of the sample in the shipping container. To be completed by the sample coordinator.

Index ID: Unique index identification number used to identify sample, in the form CSS-####. To be completed by the field team.

Sample Date: The date each sample was collected, in the form MM/DD/YY. To be completed by the field team.

Sample Time: NA. To be completed by the field team.

Sample Matrix: The matrix of each sample collected, A = Air and D = Dust. To be completed by the field team.

Project-Specific Modification

Sample Type: Sample type of each sample collected will be NA. To be completed by the field team.

Volume/Area: Specific to air and dust samples. To be completed by the field team.

Analysis Request: Analysis of air samples collected: PCM/AHERA/ISO 10312. Analysis of dust samples collected: ISO 10312

Comments: Any pertinent information regarding the sample.

Sample Received by Lab: To be checked by the sample custodian at the laboratory upon receipt of the samples to confirm presence of each sample on the COC record.

Total Number of Samples: Total number of samples on the COC form. To be completed by the field team.

Additional Comments: Any additional comments that relate to samples on the COC form (i.e., turn around times). To be completed by the field team or sample coordinator.

Relinquished by: (1) Signed by field team member that relinquishes samples to sample coordinator and company of person relinquishing samples to sample coordinator (i.e., CDM). Date of relinquish shall be in the form MM/DD/YY and time shall be in military time. (2) Additional relinquished by lines to be completed following standard sample custody procedures.

Received by: (1) Signed by sample coordinator that receives samples from the sampling team and company of person accepting samples from the field teams (i.e., CDM). Date and time of acceptance should be the same as date and time of relinquish. (2) Additional received by lines to be completed following standard sample custody procedures.

Sample Condition upon Receipt: Will reflect the condition of samples at the relinquish time (i.e., accept ok or not acceptable with an explanation). To be completed by the person receiving samples.

Page ___ of ___: Sequential page number of the entire COC set sent to the laboratory. To be completed by the sample coordinator.

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Libby Asbestos Investigation

No. 000000

Send to:		
, Region VIII		
U.S. Environmental Protection Agency, Region VIII	999 18th Street, Suite 300	Jenver, CO 80202-2413

			 	_	Ţ		_		 	
Shipped	Sample	by Lab	C		C					C
via: ☐ hand delivery ☐ shipped		Comments								
l Action CSS		Analysis Request*								
II Remova	Volume (L) or Area	(cm²)								
1 Phase	Sample Type						•			
(circle 1): Phase I Phase II Removal Action CSS	Sample Matrix (S=Sol; W=Water, 0=Due: A=Air B=Bulk	Insulation)								
Project (cir	Sample									
	Sample	Date								
		Index ID								
	Sample Placed	in Cooler/Bag	ם			ם		ם	ם	

*Phase I: Air: preparation method EPA/540/2-90/005a, analytical method PCM (by NIOSH 7400), TEM (by ISO 10312 and AHERA). Dust: preparation method ASTM D5755-95, analytical method ISO 10312. Solid PLM: preparation and analysis by ISSI-LIBBY-01/NIOSH 9002. Soil IR: preparation and analysis by ISSI-LIBBY-01/NIOSH 9002.

method ISSI-LIBBY-01/ISO 10312. Phase II: Personal Air Insulation and Soil: PLM. Dust Samples: TEM (by ISO 10 K-Ray Spectroscopy, Revison 0, July 11, 2000; Soil IR: pr	method ISSI-LIBBY.01/ISO 10312. Phase II. Personal Air, Stationary Air. PCM (by NIOSH 7400), TEM (by Modified ISO 10312 – Phase 2 QAPP, approved 2/01), or TEM (AHERA) method. Bulk Insulation and Soil: PLM. Dust Samples: TEM (by ISO 10312). CSS: Soil SEM: preparation by ISSI-LIBBY-01, analytical method Asbestos Analysis of Soil by Scanning Microscopy and Energy Dispersive X-Ray Spectroscopy, Revison 0, July 11, 2000; Soil IR: preparation by ISSI-LIBBY-01, analytical method ISSI-LIBBY-02; Water: preparation by EPA 600/4-84-034, analytical method ISO 10312.
Total Number of Samples	END OF SUBMITTAL
Additional Comments:	

Sample Condition upon Receipt	Sample Condition upon Receipt	Sample Condition upon Receipt
Date/Time	Date/Time	Date/Time
Received by (Signature and Company)	Received by (Signature and Company)	d Company) Date/Time Received by (Signature and Company) Date/Time Sample Concluded in scalability and in scalability of the second in scalability of the scalability of the second in scalability of the scalability of th
Relinquished by (Signature and Company) Date/Time	Relinquished by (Signature and Company) Date/Time	Relinquished by (Signature and Company) Date/Time May 9, 2002 Conies: Pink - Retained by Sample

Included in analytical report Retained by laboratory; white

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Prepared: David O. Johnson

Technical Review: Jackie M

QA Review: Doug Updike

Approved:

gnature/Date

Issued:

Kignature/Date

1.0 OBJECTIVE

Due to the evidentiary nature of samples collected during environmental investigations, possession must be traceable from the time the samples are collected until their derived data are introduced as evidence in legal proceedings. To maintain and document sample possession, sample custody procedures are followed. All paperwork associated with the sample custody procedures will be retained in CDM Federal Programs Corporation (CDM Federal) files unless the client requests that it be transferred to them for use in legal proceedings or at the completion of the contract.

Note: Sample custody documentation requirements vary with the specific EPA region or client. This SOP is intended to present basic sample custody requirements, along with common options. Specific sample custody requirements should be presented in the project-specific quality assurance (QA) project plan or project-specific modification or clarification form (See Section U-1).

2.0 BACKGROUND

2.1 Definitions

<u>Sample</u> – A sample is material to be analyzed that is contained in single or multiple containers representing a unique sample identification number.

Sample Custody – A sample is under custody if:

- 1. It is in your possession.
- 2. It is in your view, after being in your possession.
- 3. It was in your possession and you locked it up.
- 4. It is in a designated secure area.

<u>Chain-of-Custody Record</u> – A chain-of-custody record is a form used to document the transfer of custody of samples from one individual to another.

<u>Custody Seal</u> - A custody seal is a tape-like seal that is part of the chain-of-custody process and is used to detect tampering with samples after they have been packed for shipping.

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Sample Label – A sample label is an adhesive label placed on sample containers to designate a sample identification number and other sampling information.

Sample Tag – A sample tag is attached with string to a sample container to designate a sample identification number and other sampling information. Tags may be used when it is difficult to physically place adhesive labels on the container (e.g., in the case of small air sampling tubes).

3.0 RESPONSIBILITIES

Sampler – The sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched.

Field Team Leader (FTL) – The FTL is responsible for ensuring that strict chain-of-custody procedures are maintained during all sampling events. The FTL is also responsible for coordinating with the subcontractor laboratory to ensure that adequate information is recorded on custody records. The FTL determines whether proper custody procedures were followed during the fieldwork and decides if additional samples are required.

Field Sample Custodian – The field sample custodian, when designated by the FTL, is responsible for accepting custody of samples from the sampler(s) and properly packing and shipping the samples to the laboratory assigned to do the analyses. A field sample custodian is typically designated only for large and complex field efforts.

4.0 REQUIRED SUPPLIES

- Chain-of-custody records (applicable client or CDM Federal forms)
- Custody seals
- Sample labels or tags
- Clear tape

5.0 PROCEDURES

5.1 Chain-of-Custody Record

This procedure establishes a method for maintaining custody of samples through use of a chain-of-custody record. This procedure will be followed for all samples collected or split samples accepted.

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Field Custody

1. Collect only the number of samples needed to represent the media being sampled. To the extent possible, determine the quantity and types of samples and sample locations prior to the actual fieldwork. As few people as possible should handle samples.

2. Complete sample labels or tags for each sample, using waterproof ink.

Transfer of Custody and Shipment

- 1. Complete a chain-of-custody record for all samples (see Figure 1 for an example of a chain-of-custody record. Similar forms may be used when requested by the client). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the sample custodian in the appropriate laboratory.
 - The date/time will be the same for both signatures when custody is transferred directly to another
 person. When samples are shipped via common carrier (e.g., Federal Express), the date/time
 will not be the same for both signatures. Common carriers are not required to sign the chain-ofcustody record.
 - In all cases, it must be readily apparent that the person who received custody is the same person who relinquished custody to the next custodian.
 - If samples are left unattended or a person refuses to sign, this must be documented and explained on the chain-of-custody record.

NOTE: If a field sample custodian has been designated, he/she may initiate the chain-of-custody record, sign and date as the relinquisher. The individual sampler(s) must sign in the appropriate block, but does (do) not need to sign and date as a relinquisher (refer to Figure 1).

- 2. Package samples properly for shipment and dispatch to the appropriate laboratory for analysis. Each shipment must be accompanied with a separate chain-of-custody record.
- 3. Include a chain-of-custody record identifying its content in all shipments (refer to Figure 1). The original record will accompany the shipment, and the copies will be retained by the FTL and, if applicable, distributed to the appropriate sample coordinators. Freight bills will also be retained by the FTL as part of the permanent documentation.

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Figure 1 EXAMPLE CDM Federal Chain-of-Custody Record

CDM Federal Programs Corporation

125 Maiden Lane, 5th Floor New York, NY 10038 (212) 785-9123 Fax: (212) 785-6114 CHAIN OF CUSTODY RECORD

						LABORATORY AND ADDRESS								DATE SHIPPED			
PROJECT NAME/LOCATION							LAB CONTRACT:								AIRBILL NO.		
MEDIA TYPE													т	┰┙	1		
1. Surface Water			HCI.	IVATIVES			MPLE Grab	TYPE								1	
2. Groundwater), pH <2					[_	{					1		
3. Leachate				I, pH >12					हु ह		1 1		1	1	1		
4. Field QC	4. H2SO4, pH <2							5 5									
5. Soll/Sediment	nt 5. Zinc Acetate, pH >9								is è		1 1		1	ł	ł	}	
6. Oil	6. Ice Only								S 8		1 1		ı		1	i	
7. Waste				reserved	•				S E		1 1		1		ł	1	
3. Other		8.	Other						ANALYSES (List no. of containers submitted)						1]	
SAMPLE	L	ABOR/	TORY	PRESER-	MEDIA	SAMPLE	19	T HWE	l - I				1			_	
LOCATION		SAM		VATIVES	TYPE	TYPE	DATE	SAMPLED		1	1 1		1	1	1		emarks I ii Ms/Ms
NO		NUM	SER	ADDED				 				_	 	├	+-	(1102	
<u>l.</u> <u>2</u>									\vdash					├-	┼	<u> </u>	
<u>2. </u>							L	 			-++		+	 	+		
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5.	$\neg +$										_	+	1	\vdash	 -		
3.							<u> </u>					1		\vdash	_		
7.																	
3.																	
).																	
10.															<u> </u>		
SAMPLER SIGNATURES:																	
RELINQUISHED BY:	DAT	ETIME	RECE	VED BY:		DATE/T		ELINQUISHE	DBY:		DATE/TIME		RECEIVE	D BY:			DATE/TIM
SIGN)	\neg	(5104)			7	rs	IGN)			7	(SPGN)						
RELINQUISHED BY:	DAT	E/TIME	RECEI	VEO BY:		DATE/T		ELINQUISHE	DBY:		DATE/TIME		RECEIVE PRINT)	ED BY:			DATE/TIM
SIGN)	\neg	1	(SIGN)			7	13	iGN)			7	1	SIGNO				
OMMENTS:												_				****	

DISTRIBUTION: White and yellow copies accompany sample shipment to laboratory; yellow copy retained by laboratory. Pink copy retained by samplers.

1/98

NOTE: If requested by the client, different chain-of-custody records may be used. Copies of the template for this record may be obtained from the Fairfax Graphics Department.

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Procedure for Completing CDM Federal Example Chain-of-Custody Record (Refer to Figure 1.)

The following procedure is to be used to fill out the CDM Federal chain-of-custody record. The record is provided herein as an example chain-of-custody record. If another type of custody record (i.e., provided by the EPA contract laboratory program or a subcontract laboratory) is used to track the custody of samples, the custody record should be filled out in its entirety.

- 1. Record project number.
- 2. Record FTL for the project (if a field sample custodian has been designated, also record this name in the "Remarks" box).
- 3. Record the name and address of the laboratory to which samples are being shipped.
- 4. Enter the project name/location or code number.
- 5. Record overnight courier's airbill number.
- 6. Record sample location number.
- 7. Record sample number.
- 8. Note preservatives type and reference number.
- 9. Note media type (matrix) and reference number.
- 10. Note sample type.
- 11. Enter date of sample collection.
- 12. Enter time of sample collection in military time.
- 13. When required by the client, enter the names or initials of the samplers next to the sample location number of the sample they collected.
- 14. List parameters for analysis and the number of containers submitted for each analysis.
- 15. Enter MS/MSD (matrix spike/matrix spike duplicate) if sample is for <u>laboratory</u> quality control or other remarks (e.g. sample depth).
- 16. Sign the chain-of-custody record(s) in the space provided. All samplers must sign each record.
- 17. If sample tags are used, record the sample tag number in the "Remarks" column.
- 18. Record date shipped.
- 19. The originator checks information entered in Items 1 through 16 and then signs the top left "Relinquished by" box, prints his/her name, and enters the current date and time (military).

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20. Send the top two copies (usually white and yellow) with the samples to the laboratory; retain the third copy (usually pink) for the project files. Retain additional copies for the project file or distribute as required to the appropriate sample coordinators.

21. The laboratory sample custodian receiving the sample shipment checks the sample label information against the chain-of-custody record. Sample condition is checked and anything unusual is noted under "Remarks" on the chain-of-custody record. The laboratory custodian receiving custody signs in the adjacent "Received by" box and keeps the copy. The white copy is returned to CDM Federal.

5.2 Sample Labels and Tags

Unless the client directs otherwise, sample labels or tags will be used for all samples collected or accepted for CDM Federal projects.

- 1. Complete one label or tag with the information required by the client for each sample container collected. A typical label or tag would be completed as follows (see Figure 2 for example of sample tag; labels are completed with the equivalent information):
 - Record the project code (i.e., project or task number).
 - Enter the station number (sample number) if applicable.
 - Record the date to indicate the month, day, and year of sample collection.
 - Enter the time (military) of sample collection.
 - Place a check to indicate composite or grab sample.
 - Record the station (sample) location.
 - Sign in the space provided.
 - Place a check next to "yes" or "no" to indicate if a preservative was added.
 - Place a check under "Analyses" next to the parameters for which the sample is to be analyzed. If the desired analysis is not listed, write it in the empty slot. Note: Do not write in the box for "laboratory sample number."
 - Place or write additional relevant information under "Remarks".
- 2. Place adhesive labels directly on the sample containers. Place clear tape over the label to protect from moisture.
- 3. Securely attach sample tags to the sample bottle. On 80 oz. amber bottles, the tag string may be looped through the ring style handle and tied. On all other containers, it is recommended that the string be looped around the neck of the bottle, then twisted and re-looped around the neck until the slack in the string is removed.

SOP 1-2

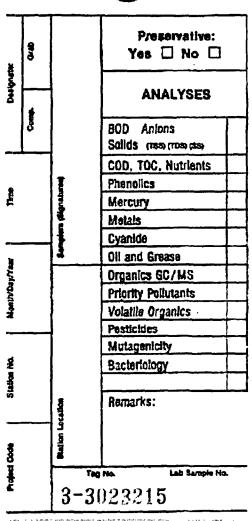
Revision: 3

Date October 12, 2001

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Figure 2
EXAMPLE Sample Tag





NOTE: Equivalent sample labels or tags may be used.

SOP 1-2

Revision: 3

Date October 12, 2001

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5.3 Custody Seals

Custody seals must be placed on the shipping containers (e.g., picnic cooler) prior to shipment. The seal should be signed and dated by a field team member.

Custody seals may also be placed on individual sample bottles. Check with the client or refer to EPA regional guidelines for direction.

5.4 Sample Shipping

The CDM Federal standard operating procedure listed below defines the requirements for packaging and shipping environmental samples.

CDM Federal SOP 2-1, Packaging and Shipping of Environmental Samples

6.0 RESTRICTIONS/LIMITATIONS

Check with the EPA region or client for specific guidelines. If no specific guidelines are identified, this procedure should be followed.

For EPA Contract Laboratory Program (CLP) sampling events, combined chain-of-custody/traffic report forms or other EPA-specific records may be used. Refer to regional guidelines for completing these forms.

The EPA FORMS II LiteTM software may be used to customize sample labels and custody records when directed by the client or the CDM Federal project manager.

SOP 1-2

Revision: 3

Date October 12, 2001

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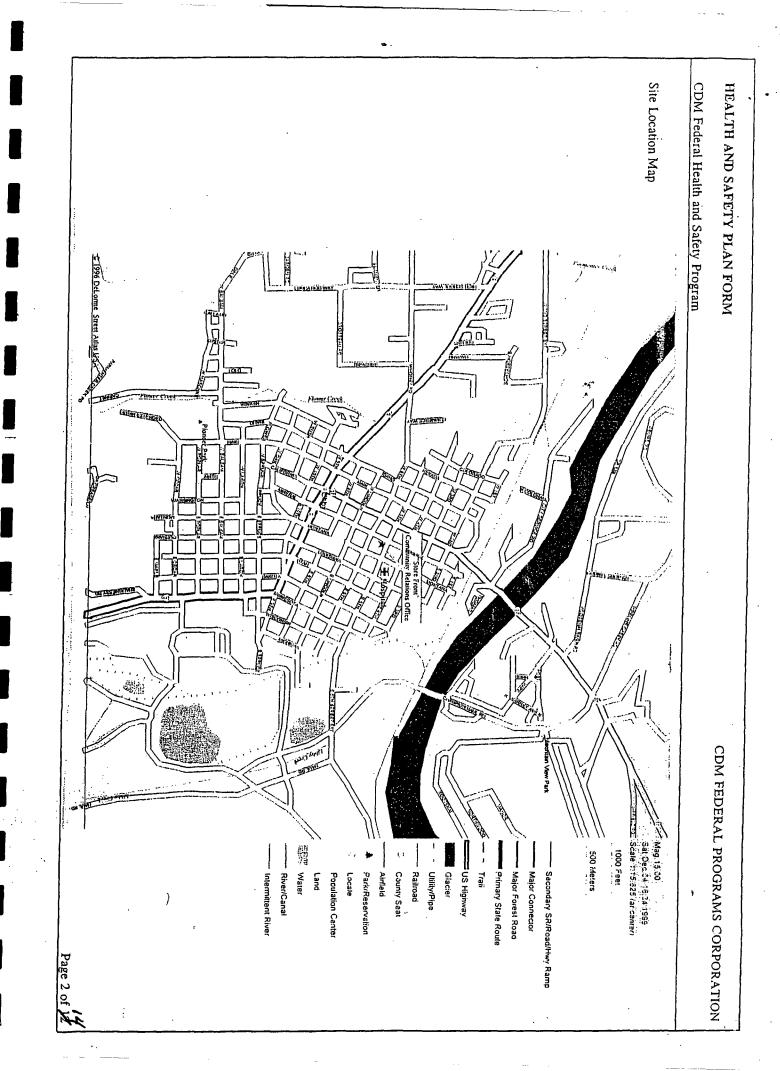
7.0 REFERENCES

- U.S. Environmental Protection Agency, EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, EPA/600/R-98/018, February 1998, Section B3.
- U.S. Environmental Protection Agency, National Enforcement Investigations Center, Multi-Media Investigation Manual, EPA-330/9-89-003-R, Revised March 1992, p.85.
- U.S. Environmental Protection Agency, Contract Laboratory Program (CLP), Guidance for Field Samplers, EPA-540-R-00-003, Draft Final, June 2001, Section 3.2.
- U.S. Environmental Protection Agency, FORMS II Lite™ User's Guide, March 2001
- U.S. Environmental Protection Agency, Region IV, Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, May 1996, Section 3.3.
- U.S. Army Corps of Engineers, Requirements for the Preparation of Sampling and Analysis Plan, EM 200-1-3, February 2001, Appendix F.

Appendix G
Health and Safety Plan

Health and Safety/Plan Form	n Form — Region 8.—	otection Agency on 8:-	CDM:Fe	CDM Federal Programs Corporation
			Project Document No.:	int No.:
Project Name	Libby Asbestos Project	Work Assignment No.	Task Orders 9, 13, 16, 18, 19, and 20	, 19, and 20 Region 8
Job Site Address	501 Mineral Avenue	Client	US DOT Volpe National T	n Systems
	Libby, Montana 59923	Project	Libby, Montana - Asbestos Removal	s Removal
Site Contact	Dave Schroeder	EPA Client Contact	Paul Peronard, EPA OSC; Jim Christiansen,	; Jim Christiansen, EPA RPM
Phone No.	406-293-8595 or 406-293-3568	Phone No.	303-312-6808	
■ Amendment No. 5	to Existing Approved HSP - Date Existing Approved HSP	8-24-2000		
Objectives of Field Work:	ត	Type: Check as many a	ny as applicable	
The purpose of this sampling effort is to acc more comprehensive environmental exposu and extent of exposure to toxicologically rel construction oversight of remedial activities	The purpose of this sampling effort is to acquire information useful for the design of more comprehensive environmental exposure investigations to assess the magnitude and extent of exposure to toxicologically relevant asbestos fibers and to provide construction oversight of remedial activities.	☐ Active ■ Inactive ■ Secure ☐ Unsecure	□ Landfill ■ Uncontrolled ■ Industrial □ Recovery	☐ Unknown☐ Military■ Other specify:Closed vermiculite mine
Remedial activities include excava of asbestos contaminated soils, by decontamination confirmatory soil activities at the Libby project sites	Remedial activities include excavation of test pits, excavation, handling and trucking of asbestos contaminated soils, building demolitions, equipment and personnel decontamination confirmatory soil sampling, air sampling, dust sampling, and related activities at the Libby project sites.	T Lincoada abaca	U vvaii niela	
Description and Features: Summarize below. Incl The town of Libby is located in the northwest corne in Libby, Montana. Vermiculite is used in various b containing material (ACM) from the following sites:	Description and Features: Summarize below. Include principal operations and unusual features (containers, buildings, dikes, power lines, hills, slopes, river) The town of Libby is located in the northwest corner of Montana. According to historical mining records, 80 percent of the world's vermiculite came from the Zonolite Mountains in Libby, Montana. Vermiculite is used in various building materials and textiles. The scope of this health and safety plan will address the processes of remediating asbestos-containing material (ACM) from the following sites:	tures (containers, buildings, nining records, 80 percent be of this health and safety	dikes, power lines, hills, slope of the world's vermiculite car plan will address the proce	es, river) ime from the Zonolite Mountains sses of remediating asbestos-
W. R. Grace Screening Plant KDC Development Properties Libby High School, Junior High School Residential Locations Export Plant Stimson Lumber Burlington Northern/Santa Fe Railroad	W. R. Grace Screening Plant KDC Development Properties Libby High School, Junior High School, and Elementary Residential Locations Export Plant Stimson Lumber			
These operations will invo	These operations will involve removal action oversight, soil sampling, and residential air sampling.	sampling.		
Surrounding Population:	Residential Industrial Rural Urban	☐ Other:		Page 1 of 15

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Health and Safety Plan Form

Environmental Protection Agency — Region 8 —

CDM Federal Programs Corporation

History: Summarize below. In addition to history, include complaints from public, previous agency actions, known exposures or injuries, etc.

the world's vermiculite came from Libby. The W.R. Grace Company, which owned the mine for 30 years, closed it in 1990 and sold the property 4 years later. Other firms used the material to make building boards and roofing materials. Processing the material was a straightforward process. The vermiculite ore was stripped from the mine and expansion plant where it was run through ovens at about 2,000 degrees, causing it to expand to 15 times its original size. In 1939, Zonolite merged with another company mining at the hauled in trucks to a mill, where it was separated into various commercial sizes through a screening system. Some of the ore was shipped untouched. Other material was sent to an Edward Alley. In 1925, Great Northem Railroad shipped the first boxcar of "Zonolite" from Libby to an Ohio company that used it to insulate bank vaults, office safes, and filing cabinets. Asbestos was released into the air during more than 6 decades of vermiculite mining at Zonolite Mountain, three miles east of Libby. The Zonolite Mine began operation in 1924 by owner Through the '60s, '70s, and '80s, millions of tons of vermiculite ore was hauled by rail to Grace plants and other companies in 30 states and 6 foreign countries. At one time, 80 percent of bottom of the hill that eventually became known as the Zonolite Co. In 1963, the company was sold to W.R. Grace and Co. who expanded the operation and increased production.

■ Cold Stress attach guidelines ☐ Inorganic Chemicals ☐ Explosive/Flammable ☐ Organic Chemicals ☐ Oxygen Deficient ■ Motorized Traffic ☐ Radiological ■ Heavy Machinery: ■ Biological: snakes, spiders ■ Slips, Trips and Falls ☐ Other Specify: Inhalation of particulate matter	Hazards of Concern: ■ Heat Stress attach guidelines ■ Noise:	*Based on available information there is no evidence that this site has received or used radioactive substances. None are described in facility processes.		Waste Types: ☐ Liquid ■ Solid ☐ Sludge ☐ Gas ☐ Unknown ☐ Other Specify: Waste Characteristics: Check as many as applicable. Work 2
dismantled. Raw vermiculite was graded, sorted, and stored at the screening plant and conveyed to a railroad loading area. Vermiculite was heated or "popped" at the export plant. The vermiculite product was then loaded onto railroad cars for distribution. Waste piles of potential asbestos-containing material (ACM) remain onsite at present. Potential ACM was distributed in the Libby area as home insulation, garden amendments, and fill dirt for a number of sites, including schools.	Principle Disposal Methods and Practices: Summarize below: The mine on Zonolite Mountain is now dormant and most of the buildings have been	in close proximity to removal activities, sampling areas, and residences. The contamination reduction zone will include an equipment and/or personal decontamination station. The support zone will be considered the 10-foot perimeter around support vehicles.	in terms onsite personnel will recognize. Work zones will be established during sampling activities. The exclusion zone will be	Specify: Work Zones: Describe the Exclusion, Contamination Reduction, and Support Zones

Page 3 of 15

Hazardous Material Summ	Hazardous Material Summary: Circle waste type and estimate amounts by category	stimate amounts by category			
Chemicals Amounts/Units:	Solids Amounts/Units:	Sludges Amounts/Units:	Solvents Amounts/Units:	Oils Amounts/Units:	Other Amounts/Units:
□ Acids	□ Flyash	□ Paint	☐ Halogenated (chloro, bromo)	□ Oily Wastes	☐ Laboratory
☐ Pickling Liquors	Asbestos	☐ Pigments	☐ Solvents	☐ Gasoline	☐ Pharmaceutical
☐ Caustics	■ Milling/Mine Tailings	☐ Metal Sludges	☐ Hydrocarbons	☐ Diesel Oil	☐ Hospital
☐ Pesticides	☐ Ferrous Smelter	☐ POTW Sludge	☐ Alcohols	☐ Lubricants	☐ Radiological
□ Dyes/Inks	☐ Non-ferrous Smelter	☐ Aluminum	☐ Ketones	□ PCBS	☐ Municipal
□ Cyanides	☐ Metals:	☐ Distillation Bottoms	□ Esters	☐ Polynuclear Aromatics	☐ Construction
☐ Phenois		-	☐ Ethers		☐ Munitions
☐ Halogens	□ Other	□ Other	□ Other	□ Other	☐ Other
□ Dioxins	Specify: Fe, Mn, Ni, Cd, Zn, Pb, Cu, Ag, Cr	Specify:	Specify: Trichloroethene (TCE), Tetrachloroethylene (PCE), cis-1,2 dichloroethene (DCE), trans-1,2 DCE	Specify:	Specify:
■ Other					
Specify: Arsenic dissolved in groundwater					
			*		
Overall Hazard Evaluation: High Justification: Air sampling results from taken by all site personnel to avoid un	i: High Medium Lessing High Medium Lessing Results from last year's activition avoid unnecessarily agitating completed as obtained	Overall Hazard Evaluation: High Medium Low Unknown (Where tasks have different his Justification: Air sampling results from last year's activities (2000) and to date (2001) have indicated low taken by all site personnel to avoid unnecessarily agitating suspect materials. PPE prescriptions are adequated and information will be completed as obtained	ks have different hazards, eval nave indicated low asbestos co criptions are adequate to addr	Overall Hazard Evaluation: High Medium Low Unknown (Where tasks have different hazards, evaluate each. Attach additional sheets if necessary) Justification: Air sampling results from last year's activities (2000) and to date (2001) have indicated low asbestos concentrations (< 10 percent of PEL/TWA). Care will be taken by all site personnel to avoid unnecessarily agitating suspect materials. PPE prescriptions are adequate to address current potential hazards. Review of recently acquired data and information will be completed as obtained	eets if necessary) 'EL/TWA). Care will be review of recently acquire
data and information will be completed as obtained					
data and information will be Fire/Explosion Potential:	□ High □ Medium ■ Low	/ □ Unknown			

				5	personal air monitoringambient/perimeter reoccupancycutting hole in ceiling - 30 minute excursion	= personal air monitoring = ambient/perimeter reoc = cutting hole in ceiling -	::*
	› پَرْ week)	ay or 40-hour working v	ıl 8-hour working da	/A) y ACGIH) tration for a norma	parts per million Short Term Exposure Limit (15 minute TWA) Threshold Limit Values (Recommended by ACGIH) Time-Weighted Average (Average concentration for a normal 8-hour working day or 40-hour working week) micrograms per kilogram micrograms per Liter		STEL TLV TWA µg/kg
		by law)	andards enforced t	and Health ation ished workplace st	Lower Explosive Limit milligrams per cubic meter Not established National Institute for Occupational Safety and Health Occupational Safety and Health Administration Permissible Exposure Limit (OSHA-established workplace standards enforced by law)	11 11 11 11 11 11	mg/m³ NE NIOSH OSHA PEL
			enforced by law)	ມstrial Hygienists າ (NIOSH standard	= American Conference of Government Industrial Hygienists = Human carcinogen = Chemical Abstract Service = Immediately Dangerous to Life and Health (NIOSH standard enforced by law)	エ	ACGIH CA CAS IDLH
		·					
N/A	Irritated eyes, mucous membranes, and respiratory system	1.0 f/cc	N/A	0.1 f/cc*/ 0.01 f/cc**	4.481 f.cc***	stos	Asbestos
N/A	Irritated eyes, mucous membranes, and respiratory system	1.0 f/cc	N/A	0.1 f/cc*/ 0.01 f/cc**	5 to 7 percent in the waste piles	stos	Asbestos
Photoionization Potential	Symptoms/Effects of Acute Exposure	Excursion Limit (≤30 minutes)	IDLH ppm or mg/m³ (specify)	PEL/TLV ppm or mg/m³ (specify)	Highest Observed Concentration (specify units and media)	Known Contaminants	Kno
ams Corporation	CDM Federal Programs Corporation	Environmental Protection Agency - Region 8 -	nvironmental Protecti – Region 8 –	Ē	an Form	Health and Safety Plan Form	H.

Page 5 of 15

 	Staff						
	Staff						
9	CA-R		100	+	ODW Cociai	3000	
			Vac		CDM Federal -	Brian Scott Pyles	Brian S
	Field Team Leader	Field To	Yes		CDM Federal -	/ Cook	Tommy Cook
	Sample Coordinator	Sample	Yes		CDM Federal -	eller	Terry Keller
	Field Team Leader	Field To	Yes		CDM Federal -	arren	Dee Warren
	Site H&S Officer	Site H	Yes		CDM Federal -	Noel Anderson	Noel Ar
	Safety Coordi	Site Health and Safety Coordinator	Yes		CDM Federal -	Douglas J. Updike	Dougla
Onsite Involvement	Work Assignment Manager	Work Assig	Yes		CDM Federal	Dave Schroeder	Dave S
	Responsibilities	Respo	CDM Federal Health Clearance	CDM Federal	Firm/Region		Name
					subcontractors)	Personnel and Responsibilities (Include subcontractors)	Person
Date: 2002			Non-Intrusive				
rea Hazard Risk: LOW	Exit Area	Level D - Modified	Intrusive		gs	Dust/Air Sampling - Commercial Buildings	8 Du
Date: 2001			Non-Intrusive				
Modified Hazard Risk: LOW	Level C - Modified	Level D - Modified	Intrusive		npling	Direct Push Technology (DPT) Soil Sampling	7 Dir
Date: 2001			Non-Intrusive				ĺ
rea Hazard Risk: LOW	Exit Area	Level C - Modified	Intrusive	den rototilling	io 4 - Sampling during gar	Residential Dust/Air Sampling - Scenario 4 - Sampling during garden rototilling activities	6 Re
Area Date: 2001	or Exit Area		Non-Intrusive				
Modified Hazard Risk: LOW	Level C - Modified	Level D - Modified	Intrusive		-	Removal Action Oversight/Air Sampling	5 Re
Date: 2001			Non-Intrusive		9,000,	0	
Modified Hazard Risk: MED	Level B - Modified	Level C - Modified	Intrusive	ulated house	io 3 - Sampling during sim ulation_replumbing_efc.)	Residential Dust/Air Sampling - Scenario 3 - Sampling during simulated house remodeling activities (e.g., removing insulation, replumbing, etc.)	4 8 9
Date: 2001			Non-Intrusive		9).	g cooping	
rea Hazard Risk: LOW	Exit Area	Level C - Modified	Intrusive	active household	io 2 - Air sampling during : nd dusting)	Residential Dust/Air Sampling - Scenario 2 - Air sampling during active household activities (e.g. yacuuming sweeping and dusting)	3 Re
Area Date: 2001	or Exit Area		Non-Intrusive		Scorps active cicarinis).		1
Nodified Hazard Risk: LOW	Level C - Modified	Level D - Modified	Intrusive	outine	io 1 - Air sampling during i	Residential Dust/Air Sampling - Scenario 1 - Air sampling during routine household activities (e.g., all activities excent active cleaning)	2 Re
Date: 2001			Non-Intrusive	-			
rea Hazard Risk: LOW	Exit Area	Level C - Modified	Intrusive			Soil Sampling -	1 So
	Contingency	Primary	Туре	ures/Site	ndard Operating Proceduessary)	Task Description/specific Technique-Standard Operating Procedures/Site Location(attach additional sheets as necessary)	Task D Location
Hazard						Field Activities Covered under this Plan	Field A
CDM Federal Programs Corporation	CDN Fee		Environmental Protection Agency — Region 8—	nylronmental Protecti – Region 8 –	E	Health and Safety Plan Form	Health

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☐ Cartridge: ☐ Escape Mask: ☐ Other: Health and Safety Plan Form Respiratory: Not Needed SCBA, Airline: ☐ Goggles: **Head and Eye:** \(\text{Not Needed} \) Block A Protective Equipment: Other: □ Goggles: ☐ Face Shield: ■ Safety Glasses: **Head and Eye:** □ Not Needed ☐ Escape Mask:
☐ Other: ■ Cartridge: P100 Respiratory:

Not Needed Block C ☐ Rubber: **Boots:**

Not Needed ☐ Face Shield: Safety Glasses: ☐ APR: □ Boots: Leather steel-toed safety boots **Boots:** \square Not Needed ■ Hard Hat: ☐ SCBA, Airline: Boots: Leather steel-toed safety boots Other: Orange traffic vest APR: ☐ Overboots: Overboots: Rubber Hard Hat: Specify by task. Indicate type and/or material as necessary. Use copies of this sheet if needed Level: C -Tasks: 6 Level: D - Modified Tasks: 5 Modified ■ Tyvek Coverall:

☐ Cloth Coverall:

☐ Other: ☐ Undergloves:☐ Gloves: Nitrile or surgical/latex.☐ Overgloves: □ Undergloves: Gloves:
Not Needed ☐ Apron activities are those that occur outside of an exclusion zone. If exclusion zone Gloves: ■ Not Needed ☐ Tyvek Coverall: if needed☐ Cloth Coverall: Cotton as needed☐ Other: Long pants & long-sleeved shirt ☐ Other - specify below: ■ Gloves: Leather
□ Overgloves: □ Splash Suit: □ Splash Suit: □ Encapsulated Suit: Prot. Clothing: | Not Needed ■ Other - specify below: Level D ☐ Encapsulated Suit: Prot. Clothing: Not Needed entry is required, use contingency. Apron ■ Primary
□ Contingency Primary Contingency Primary Environmental Protection Region 8 -☐ Overboots:☐ Rubber: ☐ Hard Hat: ■ Hard F ☐ Boots: Leather steel-toed work boots **Boots:**

Not Needed ☐ Goggles: ☐ Face Shield: Safety Glasses: Head and Eye: Other: ☐ Escape Mask: □ Cartridge: Respiratory:
Not Needed
SCBA, Airline: Block D ☐ Boots: Leather steel-toed safety boots Boots: Not Needed ☐ Goggles: ☐ Face Shield: Head and Eye: Not Needed ☐ Escape Mask: Respiratory:
Not Needed
SCBA, Airline: Block B □ APR: Safety Glasses: Cartridge: P100 Overboots: Hard Hat: Other APR: |Agenc) Not Needed م مول Level: Exit Area Tasks: 6 Level: C- Modified or Exit Area Tasks: 5 ☐ Tyvek Coverall: ☐ Cloth Coverall: ☐ Other: CDM Federal Programs Corporation Gloves:

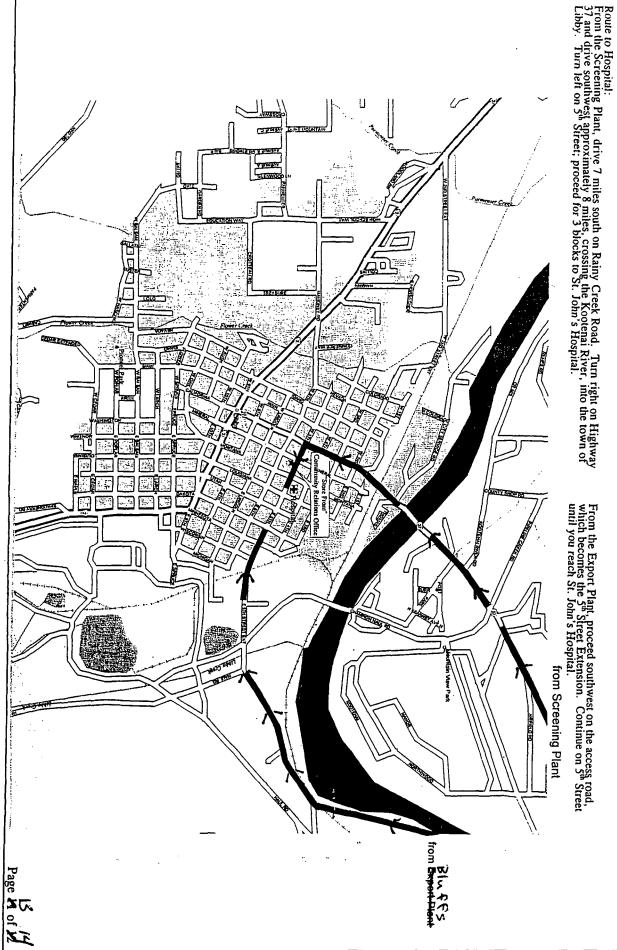
Not Needed ☐ Overgloves: ☐ Undergloves: ☐ Gloves: □ Apron require entry/egress through the ■ Other - specify below: All activities □ Overgloves: Gloves: ☐ Not Needed ☐ Other: Long pants & long-sleeved ☐ Splash Suit: ☐ Encapsulated Suit: Prot. Clothing: | Not Needed which occur within the exclusion zone ☐ Undergloves: ☐ Cloth Coverall: Cotton as needed ■ Tyvek Coverall: Splash Suit: ☐ Encapsulated Suit: Prot. Clothing: ☐ Not Needed Gloves: nitrile or surgical/latex lecon trailer. Other - specify below: Apron ☐ Primary
■ Contingency Exit Area Contingency Primary Page 9 of 15

Health and Safety Plan Form Type Combustible Gas Indicator Specify: Visible or nuisance dust Other Respirable Dust Monitor Type **Detector Tubes/Monitor** Photoionization Detector Type Radiation Survey Meter Monitoring Equipment: Specify by task. Indicate type as necessary. Attach additional sheets as necessary. and/or unusual vapors (odors) Type Flame Ionization Detector Instrument □ 11.7 eV □ 10.2 eV □ 9.8 eV □ eV 1-8 1-8 1-8 1 - 8 1-8 1 - 8 1 - 8 Task 21.0% 0₂ <21.0% 0₂ <**21.0% 0₂** <**19.5% 0₂** Specify: If team notices unusual odors, heavy dust, or irritation of the eyes or throat, they will exit area and Specify: Specify: Specify: 0-10% LEL 10-25% LEL **>25% LEL** 3X Background >2mR/hr reevaluate personnel protection. Detectable Odor Specify: Environmental Protection Agency — Region 8— Notify SHSO and CDM Federal HSM, establish REZ If odor of any kind is detected, cease Oxygen deficient; notify SHSC Oxygen normal Explosion hazard; interrupt Potential explosion hazard, notify SHSC No explosion hazard work, move to fresh air. Interrupt task/evacuate Interrupt task/evacuate task/evacuate Action Guidelines CDM Federal Programs Corporation into confined spaces where toxic gases could be concentrated is strictly forbidden. Comments (Include schedules of use) encountered during site work. Entrance If further work is necessary in the area If further work is necessary in the area If dusty conditions persist, site will be where odors are detected, personnel protection will be evaluated. abandoned and personnel protection where odors are detected, personnel Oxygen-deficient or other dangerous Toxic gases are not expected to be encountered. Entrance into confined atmospheres are not expected to be spaces where toxic gases could be concentrated is strictly forbidden. protection will be evaluated ■ Not Needed Not Needed Not Needed Not Needed Not Needed Not Needed Page 11 of 15 reevaluated

(4) remove safety glasses(5) remove Tyvek or cloth coverall, if used(6) remove respirator, if used(7) remove inner gloves Health and Safety Plan Form (1) wash overboots in soapy water and rinse(2) remove overboots or booties(3) remove gloves shower will be taken as soon as possible after leaving the *If standard field clothes are used, the clothing must be kept separate from all personal articles and washed before it is reused. Under no circumstance can soiled clothing be donned at the beginning of each work day. Any article of clothing that becomes unusually soiled during field activities must be replaced with a clean item. Hazardous Materials Inventory (Investigation-Associated Substances: All disposable PPE will be double-bagged prior to disposal (8) wash hands/face before eating/drinking field. Workers will remove protective clothing in this order: Wash well before hand to mouth contact is made. A Personalized Decontamination Decon water to be disposed onsite Containment and Disposal Method Decontamination Procedures Sulfuric Acid (H₂SO₄)
Sodium Hydroxide (NaOH)
Zinc Acetate (ZnOAc) Nitric Acid (HNO₃) Hydrochloric Acid (HCI) Preservatives Other: Ascorbic Acid □ Not Needed Not Needed Decon water to be disposed onsite (5) air dry
(6) wrap in aluminum foil for transport thoroughly decontaminated as follows: See CDM Federal SOP 4-5. All sampling equipment will be (4) thoroughly rinse with deionized water (2) potable tap water rinse(3) potable tap water rinse (1) wash and scrub with low phosphate detergent Sampling Equipment Decontamination Containment and Disposal Method Liquinox TM Methanol Mineral Spirits Acetone AlconoxTM Environmental Protection Attach MSDS Decontamination Region 8 -Other: Water Nitric Acid Isopropano Hexane Agency □ Not Needed Not Needed **Exclusion Zone Egress Decontamination** All disposable PPE will be double-bagged prior to disposal. closed in order to maintain negative pressure Note - All doors on the decon trailer must remain Containment and Disposal Method Heavy Equipment Decontamination (10) proceed to clean room, dress, and exit (4) enter decon trailer dirty room
(5) remove Tyvek coveralls
(6) enter shower and clean respirator
(7) remove respirator after thorough cleaning (8) complete personal decon (9) dry with disposable towels (2) remove overboots(3) hang safety vest and hard hat in exclusion area wash overboots Propane Pentane Methane Isobutylene Hydrogen equipment drop room Calibration Gases and Fluids CDM Federal Programs Corporation Other Other pH Standard Conductivity Standard □ Not Needed □ Not Needed Page 12 of 15

Page 13 of 15		Distance to Hospital: Variable		noval	Site: Libby, Montana, Asbestos Removal
					For: Chuck Myers, CIH
re located in the site and 4th Avenue.	nospital will vary depending on where you are located in th	Directions to the hospital will vary depending on where you are located in the site area. The hospital is located at the intersection of Louisiana and 4th Avenue.		J.	all h
niliar with location of	map (Page 13) to the hospital. Become familiar with location of ny site activities.	See the attached map (Page 13) hospital prior to any site activities	Date:		HSM Signature:
	(Attach.map with route to hospital)	Route to Hospital (Attachmap w	Date:		SHSO Signature:
911		Name of 24-Hour Ambulance:	Date: 8-29-2002		Prepared by: Doug Updike
	A	Name of Contact at Hospital: NA			Health and Safety Plan Approvals
	Avenue	Hospital Address: 350 Louisiana Avenue	ated.	tection will be reevalu	eliminate the exposure, personnel protection will be reevaluated.
911	ran Hospital	Hospital Name: St. John's Lutheran Hospital	eevaluated. In the event of epartment. If respirable dust	Police, or Sheriff's De	medical emergency, contact Hospital, Police, or Sheriff's Department. If respirable dust
		Medical Emergency - **	us to human health. If any	ncentrations dangerous	are expected to be encountered at concentrations dangerous to human health. If any
1-800-229-3674	Dr. Edward Barnes	Occupational Physician	ill leave the site and upgrade	A Federal personnel w	CDM Federal Health and Safety. CDM Federal personnel will leave the site and upgrade
1-800-525-5042		Poison Control Center	encountered. If staff observe	rdous conditions are on they will with the conditions are on the conditions are only only only on the conditions are only only on the conditions are only only only on the conditions are only only only only on the conditions are only only only only only only only only	Evacuate site if any unexpected hazardous conditions are encountered. If staff observe hazards for which they have not been prepared they will withdraw from the area and call
1-800-525-5555	Highway Patrol	State Police		QW.	Contingency Plans Summarize below.
911		Police Department - Libby			
911	•	Fire Department			
911		State Spill Line		1-800-424-9300	CHEMTREC Emergency
911	Lincoln County	Sheriff's Department	-3567	SHSO 1-406-293	
1-406-293-7881		Health Department	Chuck Myers (home) 1-703-754-0700	Chuck Myers (ho	Other (Specify) Health & Safety Mgr.
1-800-234-5677		Environmental Agency		NA	Facility Management
1-303-312-6808	Paul Peronard	EPA Contact	/004	(cell) 1-5/1-216-/004	Cnuck Myers
1-617-494-2574	John McGuiggin	DOT Contact			CDM 24-Hour Emergency
1-406-293-3567 1-816-412-3149	Noel Anderson Douglas J. Updike	Health & Safety Coordinator		1-800-424-8802	EPA Release Report No.
1-406-293-3388	Dave Schroeder	Site Manager		1-406-293-8595	Site Telephone
1-703-968-0900	Chuck Myers, CIH	Health and Safety Manager		NA	Water Supply
Phone :	Name	Emergency Contacts			Етедепсу Сопдаст
CDM Federal Programs Corporation	CDM Federal R	tal Protection Agency Region 8 ≂	Environmental Protection Agency — Region 8 —		Health and Safety Plan Form
	of the following strains along the party for the same of the same of the same strains and the same of the same strains and the same strains are same				and the state of t

CDM Federal Health and Safety Program



Health and Safety Plan Form Brian Scott Pyles Dee Warren Douglas Updike Dave Schroeder Site: Libby, Montana - Asbestos Removal The following personnel have read and fully understand the contents of this Health and Safety Plan and further agree to all requirements contained herein. Tommy Cook Terry Keller Noel Anderson Name and Responsibility CDM Federal Programs Corporation **CDM Federal Programs Corporation CDM Federal Programs Corporation** Project No.: Environmental Protection Agency
— Region 8 — Date ار مولی CDM Federal Programs Corporation Signature Page 15 of 15